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RESEARCH ARTICLE



An Investigation into the Suitability and Stability of a New Pigmented Wax-Resin Formulation for Infilling and Reintegration of Losses in Paintings

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ABSTRACT

A new Pigmented Wax-Resin (PWR) formulation for infilling and reintegration of losses in paintings is introduced and tested for its suitability and stability. It consists of a mixture of Cosmoloid H80 microcrystalline wax and Regalrez 1126 hydrogenated hydrocarbon resin with dry pigments and/or fillers. Unlike other PWR formulations, including those sold by Gamblin Conservation Colors, it does not contain beeswax. Beeswax is reported to develop bloom and to corrode copper supports. The authors share methodologies and techniques used to characterize and assess the suitability of the new formulation in terms of its physical and optical properties, and to assess its stability to fluctuating relative humidity and temperature, particularly high temperatures. The new formulation was evaluated for its workability, opacity, and flexibility and for its compatibility with a selection of varnish coatings and inpainting media. Results showed that 1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126 (by weight), mixed with pigments and/or fillers, is a viable alternative to other infilling and inpainting media. It has good optical and working properties, a suitable degree of hardness, and remains stable during fluctuations in relative humidity and temperature, as well as to temperatures as high as 70°C.

RÉSUMÉ

Une nouvelle formulation de cire-résine pigmentée (CRP) pour le masticage et la réintégration de lacunes dans les peintures est introduite et testée pour sa pertinence et sa stabilité. Elle se compose d'un mélange de cire microcristalline Cosmoloid H80 et de résine d'hydrocarbure hydrogéné Regalrez 1126 avec des pigments secs et/ou des charges. À l'encontre d'autres formulations de CRP, comme celles vendues par Gamblin Conservation Colors, elle ne contient pas de cire d'abeille. Il est rapporté que la cire d'abeille produit de l'efflorescence et corrode les supports en cuivre. Les auteurs partagent les méthodologies et les techniques utilisées pour caractériser et établir la pertinence de la nouvelle formulation en termes de propriétés physiques et optiques, et établir sa stabilité face aux variations d'humidité relative et de température, particulièrement aux températures élevées. La nouvelle formulation a été évaluée pour sa maniabilité, son opacité et sa flexibilité, et pour sa compatibilité avec une sélection de couches de vernis et de matériaux de retouche. Les résultats montrent que 1,5 mesure de Cosmoloid H80 pour 1 mesure de Regalrez 1126 (en poids), mélangées avec des pigments et/ou des charges, est une alternative viable à d'autres mastics et matériaux de retouche. Elle a de bonnes propriétés optiques et est facile à travailler, a un degré de dureté approprié, et reste stable lorsque soumise à des variations d'humidité et de température, ainsi qu'à des températures aussi élevées que 70°C. *Traduit par Isabelle Cloutier.*

RESUMO



Uma nova formulação pigmentada de resina de cera (PWR) é introduzida e testada no preenchimento e reintegração de lacunas nas pinturas para verificar a sua adequação e estabilidade. Consiste numa mistura de cera microcristalina Cosmoloid H80 e uma resina de hidrocarboneto hidrogenado Regalrez 1126 com pigmentos secos e/ou cargas. Ao contrário de outras formulações de PWR, incluindo as vendidas pela Gamblin Conservation Colors, não contém cera de abelha. Diz-se que a cera de abelha desenvolve florescimento e corroi os suportes de cobre. Os autores partilham metodologias e técnicas usadas para caracterizar e avaliar a adequação da nova formulação em termos das suas propriedades físicas e óticas, e para avaliar a sua estabilidade nas oscilações da humidade relativa e temperatura,

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Pigmented Wax-Resin; Cosmoloid H80; Regalrez 1126; pigments; inert fillers; stability; infilling; reintegration

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particularmente em temperaturas elevadas. A nova formulação foi avaliada pela sua viabilidade, opacidade e flexibilidade e pela sua compatibilidade com uma seleção de revestimentos de verniz e materiais de retoque. Os resultados mostraram que 1,5 partes do Cosmoloid H80 para 1 parte de Regalrez 1126 (em peso), misturadas com pigmentos e/ou cargas, é uma alternativa viável a outros meios de preenchimento e retoque. A formulação possui boas propriedades óticas e de viabilidade, um grau adequado de dureza, e permanece estável durante as flutuações na humidade relativa e de temperatura, bem como a temperaturas até 70°C. *Traduzido por Teresa Lança.*

RESUMEN

Se presenta una nueva formulación de Cera-Resina Pigmentada (PWR) para el estucado y reintegración cromática de pérdidas de pintura y se hacen pruebas para determinar su idoneidad y estabilidad. Consiste en una mezcla de cera microcristalina Cosmoloid H80 y resina de hidrocarburo hidrogenado Regalrez 1126 con pigmentos secos y/o medios para el estucado. A diferencia de otras formulaciones PWR, incluidas las comercializadas por Gamblin Conservation Colors, no contiene cera de abejas. Se ha informado que la cera de abejas desarrolla pasmosos o blanqueamiento y corroe los soportes de cobre. Los autores comparten las metodologías y técnicas empleadas para caracterizar y evaluar la idoneidad de la nueva formulación en cuanto a sus propiedades físicas y ópticas y para evaluar su estabilidad a las fluctuaciones de humedad relativa y temperatura, en particular a las altas temperaturas. Se evaluaron la maleabilidad, la opacidad y la flexibilidad de la nueva formulación, así como su compatibilidad con una selección de revestimientos de barniz y medios usados en la reintegración cromática de pérdidas de pintura. Los resultados mostraron que 1,5 partes de Cosmoloid H80 por 1 parte de Regalrez 1126 (en peso), mezclado con pigmentos y/o medios utilizados para el estucado, es una alternativa viable a otros medios de estucado y reintegración cromática. Tiene buenas propiedades ópticas y de maleabilidad, un grado adecuado de dureza y permanece estable durante las fluctuaciones de humedad relativa y temperatura, así como a temperaturas de hasta 70°C. Traducción: *Ramón Sánchez*; revisión: *Julia Betancor*; revisión final: *Irene Delaveris y Amparo Rueda.*

1. Introduction

1.1. Advantages of pigmented wax-resin (PWR) formulations

PWR formulations are composed of a mixture of a wax and a resin to which fillers (such as chalk) and/or pigments are added. These formulations can be used to infill and reintegrate losses in paintings, presenting several advantages (detailed below) that make them viable options alongside other materials used in paintings conservation.

A significant advantage of PWRs lies in the possibility of adding pigments to the wax-resin (WR) mixture, which enables both infilling and color reintegration in a single step. An accurate and quick color match is possible since hues do not change as the mixture transitions from warm (fluid) to cool (solid) states. In some applications, color matched PWRs may not require further treatment with varnishes since they can be polished with a soft cloth to achieve a high gloss, or rendered matte with solvent or by burnishing with matte silicone or wax coated paper (see Appendix 3). Several authors have highlighted their good adhesion properties, ease of application and ease of removability (Ruhemann 1968; Althöfer 1985; Gänssicke and Hirx 1997; McIntyre 2011; Fuster-López 2021). PWRs can be removed mechanically or with

low-aromatic solvents such as white spirits (for details on working properties see Rocha Pires et al. 2023).

Where required, PWRs, including the new formulation presented here, accept surface adjustments with glazes using different inpainting media. A more traditional method of reintegrating losses is also possible, as the WR mixture, with the addition of chalk or pigments, can be used as a neutral or toned base over which further layers of inpainting can be applied using an appropriate resin based retouching medium.

Another important advantage is that PWR infills can easily receive texture by carving with a scalpel or imprinting with textured silicone molds, as well as being sculpted. Surface features, such as impasto, brushstrokes, craquelure or canvas weave patterns, can be easily imitated.

1.2. PWR formulations: background and history of use

PWR formulations have been used to infill and reintegrate losses in paintings and other objects at least since the eighteenth century (Pernety 1757). Recipes for PWR formulations found in the literature (1757–2019) are compiled in Appendix 1, along with different application methods. The wax referred to in most

recipes is beeswax, although replacements were explored in the 1970s, when other materials such as polyethylene glycols (Althöfer 1959), ethylene acrylic acid copolymers (Gänsicke and Hirx 1997), paraffin wax (at the Stichting Restauratie Atelier Limburg/SRAL: K. Seymour, pers. comm. 2019) and, more recently, microcrystalline waxes (Kemp 2009; Vega 2016; González 2017; von Lenthe 2019), were introduced. Nevertheless, beeswax has remained in use, for example, in the 1980s (e.g., Schneider 1981; Althöfer 1985; Ruhemann 1968), in the 1990s (e.g., Garrel 1992; Vega 2016; Nicolaus 1999) and into the 2000s (e.g., Kemp 2009; Folkes and Reddington 2010; McIntyre 2011; Gamblin Conservation Colors n.d.).

If some of the recipes contain only wax (mixed with fillers/pigments), the inclusion of a resin is already mentioned by some of the earliest authors (e.g., Pernety 1757; Déon 1851; Kudrjawzew 1954; Brachert 1955). Where specified, the resin present in the recipes is either natural (dammar resin [Althöfer 1985; Nicolaus 1999; Folkes and Reddington 2010], colophony [Schneider 1981; Nicolaus 1999]), synthetic (cyclohexanone-aldehyde resins, or ketone, polyvinyl [Gänsicke and Hirx 1997], aldehyde [McIntyre 2011; Gamblin Conservation Colors n.d.], and hydrocarbon resins [Vega 2016; González 2017]) or a mixture of both (e.g., colophony and Alkydal, a mixture of equal parts of synthetic alkyd resin and linseed oil, used by Schneider in 1981). Most of these recipes include fillers (e.g., chalk, Portafill A40, fumed silica) and/or pigments which are mixed with the WR binder. Additional materials, such as oils and glues, were listed as well.

Traditionally, PWR formulations were prepared by individual conservators. However, in 2014, Robert Gamblin introduced the Gamblin Pigmented Wax/Resin (Gamblin-PWR), which consist of beeswax, Be-Square 195 microcrystalline wax, Laropal A81 resin and pigments, and are described as being “ideal for filling losses in paintings” (Gamblin Conservation Colors n.d.). This formulation resulted from research conducted by Christine McIntyre (2011), which built on the work of Frederick Wallace (1990, unpublished). McIntyre’s research was focussed on finding a suitable resin to replace Laropal K80 which was no longer being manufactured and had formed the basis of the successful PWR formulation employed at the Buffalo State College Art Conservation Program (McIntyre 2011).

1.3. Beeswax in PWR formulations

Although generally considered to be stable and chemically resistant (Čížová et al. 2019), the reported

tendency of beeswax to bloom has been a concern since the color of the infill can be altered and distorted due to the “whitish efflorescence that develops on the surface of beeswax objects under specific conditions” (Bartl et al. 2015). Even though the presence of varnish over a PWR containing beeswax has been reported to inhibit the development of bloom (von Lenthe 2019, 37, 44), these infills would not be suitable for unvarnished paintings.

The formation of bloom has been linked to temperature (T) fluctuations, as well as high relative humidity (RH) (Bartl et al. 2012, 2015). McIntyre, writing in 2011, observed that of the 10 samples which contained unbleached beeswax prepared by Wallace in 1990, 8 showed clear evidence of bloom in the form of a whitish haze. She also noted that none of Wallace’s PWR formulations without beeswax exhibited bloom (McIntyre 2011). Although McIntyre did test new formulations containing only microcrystalline waxes (Be Square 195, Multiwax W-445 and Cosmoloid H80) and Regalrez 1094 resin, she rejected these as she found that those mixtures did not pour easily, and the pigments tested did not stay suspended in the samples made (McIntyre 2011). Later, in 2016, Vega concluded that it was not possible to obtain a mixture with Laropal A81 (the resin selected by McIntyre to replace Laropal K-80), nor Laropal A101, and microcrystalline waxes without also adding beeswax (Vega 2016; González 2017). This is likely the reason why beeswax was not removed from McIntyre’s PWR formulation and is still incorporated in the Gamblin-PWR formulation.

Beeswax has also been reported to promote corrosion of copper supports used in oil paintings (Horovitz 1996; Scott 2002; Paterakis 2003). The acidic nature of beeswax, due to the presence of carboxylic groups from the fatty acids, was confirmed with acid value measurements and FTIR analyses (Vega 2016; Scott 2002).

1.4. PWR formulations without beeswax

Projects to explore replacing beeswax with a synthetic wax were initiated in the conservation training program at the NOVA School of Science and Technology (Vega 2016; Vega, Cardoso, and Carlyle 2017). Vega’s most successful PWR formulation contained a mixture of Techniwax 9426 microcrystalline wax and Regalrez 1094 hydrogenated hydrocarbon resin. To find a more readily available microcrystalline wax, a formulation to replace the Techniwax 9426 with Cosmoloid H80 was explored, in that case mixed with Regalrez 1126 (González 2017).

The development of a successful ratio of the latter mixture by Rocha Pires and its evaluation (Rocha

Pires 2021) is detailed here. A mixture of testing methods was adopted, some involving sophisticated equipment, including an environmental chamber and Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR), while other methods were studio based and more practical in nature. This approach was adopted because of limitations in accessing instrumentation used for ATSM standards and specialized laboratory equipment used in the study of waxes.

2. Methodology for testing

To find the best PWR (referred to as NOVA-PWR) containing Cosmoloid H80 wax and Regalrez 1126 resin, a study of the mixture in different proportions, together with different pigments and fillers, resulted in over 800 samples of the most promising formulations. Samples were compared directly with Gamblin-PWR which provided the gold standard for a successful PWR in terms of working properties, and a basis of comparison in terms of development of bloom. Details on the number and type of samples used in each testing regime are given in Rocha Pires 2021.

There were three main goals:

- 1) to evaluate how the materials in the NOVA-PWR (wax, resin, pigments and fillers) and their presence in different proportions could influence visual and physical properties (such as hardness, opacity/translucency and flexibility) and behavior in terms of preparation and ease of application.
- 2) to evaluate their stability, for which selected NOVA-PWR formulations (Appendix 2) were subjected to changes/fluctuations in T and RH, as well as to high T: 50°C, 60°C, and 70°C (the latter with radiant heat). Stability parameters included the development of bloom, changes or loss of texture (due to softening), and mechanical changes such as cracking. Gamblin-PWR samples were exposed to the same environmental conditions for comparison. Evaluation of visual, physical, and molecular changes was done with macro photography, digital microscopy, hardness measurements, and ATR-FTIR.
- 3) to assess the compatibility of the successful NOVA-PWR formulation with a selection of varnishes and inpainting media commonly used in conservation.

To evaluate the workability of the successful NOVA-PWR formulation, losses were infilled in two deaccessioned works at SRAL (Maastricht, The Netherlands): an eighteenth century oil painting and an acrylic

painting dating from 2002. The results are published in Rocha Pires et al. (2023).

3. Materials and methods

3.1. PWR materials and sample preparation

3.1.1. Materials

Gamblin Pigmented Wax/Resin (Gamblin-PWR), kindly supplied by Gamblin Conservation Colors, contain beeswax, BeSquare 195 microcrystalline wax, and Laropal A81 resin (McIntyre 2011; Fuster-López 2021). An unpigmented “Neutral Base,” and three colored Gamblin-PWR, “Chrome Oxide Green,” “Burnt Umber,” and “Titanium White,” were used for this research. If the “Chrome Oxide Green” and “Burnt Umber” Gamblin-PWRs were chosen because they were observed to have had developed bloom while stored inside the metallic box in which they are sold, “Titanium White” was included for being one of the most popular white pigments used either alone or in mixtures with other pigments. For direct comparison the NOVA-PWR formulations included an unpigmented neutral base and the same set of pigments.

NOVA-PWR ingredients:

- **Wax:** Cosmoloid H80 is a microcrystalline wax (Table 1) manufactured by Astor (now The International Group, Inc.) and widely available to conservators (e.g., from Kremer Pigmente GmbH & Co. KG; Talas; C.T.S; and Zeus). Cosmoloid H80 has been recommended by Velson Horie as being an inert material with a suitable degree of hardness (Horie 1990). It is also considered highly stable to chemical degradation (Matteini, Mazzeo, and Moles 2016).
- **Resin:** Regalrez 1126 (Table 1) is a synthetic low molecular weight hydrogenated hydrocarbon resin that, along with Regalrez 1094, has been widely used in conservation. While Regalrez 1094 is more commonly used as a picture varnish (De la Rie and McGlinchey 1990), Regalrez 1126 has been used as a furniture polish/varnish (Piena 2001). Regalrez 1126 was chosen due to its higher molecular weight, higher glass transition T (T_g) and higher softening point (Table 1).

Table 1. Properties of Cosmoloid H80 and Regalrez 1126.

	Molecular weight	Acid value	Melting point	T_g	Density at 21°C
Cosmoloid H80	-	0 [1]	83-94°C [1]	-	0.85-1.05 g/cm ³ [1]
Regalrez 1126	1250 g/mol [2]	0 [3]	124 [4]	67°C [2]	0.97 g/cm ³ [2]

[1] Kremer Pigmente n.d.a

[2] Kremer Pigmente n.d.b.

[3] Deduced from Vega's acidity measurements on wax-resin mixtures with Techniwax 9426 and Regalrez 1094 (Vega 2016).

[4] Labshop 2014.

Regalrez resins have been reported to be photochemically stable (De la Rie and McGlinchey 1990), UV stable (Whitten 1995), and resistant to change under accelerated environmental conditions (von der Goltz et al. 2012). Regalrez resins have high compatibility with microcrystalline waxes (Piena 2001).

- **Pigments:** Chrome oxide green (PG 17.77288), burnt umber (PBr 8.77727) and titanium white (PW 6.77891). Chosen for comparison with the Gamblin-PWRs selected and for their high thermal stability and lightfastness.
- **Fillers:** Champagne chalk (PW 18.77220), kaolin (PW 19.77004), and aluminum hydroxide (Portafill A40). Although not present in the Gamblin-PWR, fillers were added alone or in combination with pigments to change or improve visual and physical properties, i.e., providing bulk, adjusting color, transparency and gloss.

3.1.2. Sample preparation

In total, 51 different NOVA-PWR formulations were prepared following the steps below (for quantities see Appendix 2):

- 1) A weighed portion of Cosmoloid H80 was placed in a glass beaker and heated on a hot plate to $\sim 70^{\circ}\text{C}$ – 75°C (T measured with a digital thermometer, see Appendix 3).
- 2) Regalrez 1126 was ground to a fine powder in a ceramic mortar and pestle (powdering accelerated mixing and melting), and a weighed portion was added gradually to the molten wax. To facilitate incorporation, T was increased to 85° – 90°C . A magnetic stirrer was used at this stage.

- 3) After 20–25 min of heating and mixing, the wax and the resin were completely incorporated (designated WR when without pigments/fillers).
- 4) Pigments and/or fillers were weighed and incorporated gradually while stirring. Only Champagne chalk and kaolin were passed through a fine mesh strainer to remove large particles, this was not necessary for aluminum hydroxide or the pigments.
- 5) The WRs and PWRs were then either poured onto a Melinex sheet to solidify prior to being evaluated for workability; or into molds to create samples for testing.

Two types of test samples were designed without substrates:

Rectangular samples ($3.4 \times 2.3 \times 0.1$ cm) of 18 NOVA-PWR and 4 Gamblin-PWR were made by pouring PWR mixtures into the center of a diapositive frame (Figure 1a). These were prepared with glossy and matte sections (to assess the development of bloom), and a circular protrusion with sharp edges (to assess any surface changes due to elevated T) (Figure 1b).

Thick circular samples (3.5 cm diameter and 0.6 cm thick) of 18 NOVA-PWR and 4 Gamblin-PWR were prepared for hardness measurements before, during and after exposure in the environmental chambers (Figure 1c).

In addition, it was considered essential to apply the different NOVA-PWR and Gamblin-PWR infills in a more realistic context, on canvas substrates. Two different sets were prepared:

- Even layers of NOVA-PWRs with three different fillers were applied onto 18 pieces of linen canvas ($8 \text{ cm} \times 6 \text{ cm}$) previously sized with Plextol B500 (Figures 2a–c) to establish their flexibility in bending trials.

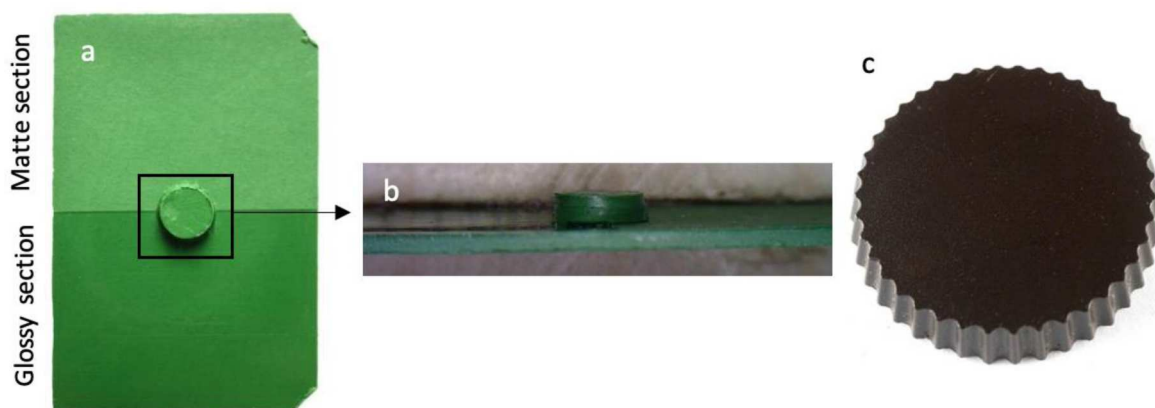


Figure 1. (a) Rectangular sample ($3.4 \text{ cm} \times 2.3 \text{ cm} \times 0.1 \text{ cm}$) photographed from above; (b) Rectangular sample photographed from the side, with focus on the circular protrusion (with a thickness of approximately 0.2 cm); (c) Circular sample (3.5 cm in diameter and 0.6 cm thick) for hardness measurements.

These samples were not subjected to any environmental trial.

- 18 NOVA-PWRs and material from 4 Gamblin-PWR sticks were applied to losses in three model paintings (Reis 2011) (Figure 2d). Losses were created with a ~ 1 cm hole punch (see Appendix 3) leaving only the animal-glue sized canvas present. PWRs were leveled to the surface of the painting resulting in ~ 0.5 mm thick infills (Figure 2e). One painting was kept as control, the other was exposed to fluctuating RH and the third to fluctuating T. The infills on the painting to be exposed to fluctuating T were scored with horizontal and vertical lines to allow assessment of change due to softening at high T (Figure 2f).

3.2. Methods to characterize and assess suitability

NOVA-PWRs were evaluated in terms of their optical and physical properties and compared with the Gamblin-PWR, in order to select successful formulations for further testing.

Unpigmented WR mixtures were assessed for workability and hardness. Samples were prepared from the most promising with the three pigments and the three

fillers. Sets were then tested, along with Gamblin-PWR samples, for workability, hardness, and opacity based on the following criteria:

Workability: Assessed experientially to establish the feasibility and ease of application in thin and thick layers and for creating three-dimensional features (impasto). T required for melting each formulation was assessed by closely observing changes while slowly increasing heat on a hot plate (see Appendix 3).

Hardness: The degree of hardness during application was assessed by feel and measured with a digital Shore D durometer (see Appendix 3) to compare different formulations. A Shore D durometer is used in the “Standard Test Method for Rubber Property – ASTM D2240 – 15” (ASTM 2021) which, however, requires an expensive set of calibration materials not available for this study. The hardness values given are, therefore, not fixed and are only meant to be used for comparison between the different formulations.

Opacity: Tests were adapted from the “International Standard for the Determination of Hiding Power in Paints and Varnishes:” ISO 6504-3:2019 (ISO 2019). Equipment to apply WR mixtures in a fixed thickness were not available, nor was instrumentation to measure opacity with reflectometry (ASTM D2805-11:2018; ASTM 2018). In addition, there was only a limited

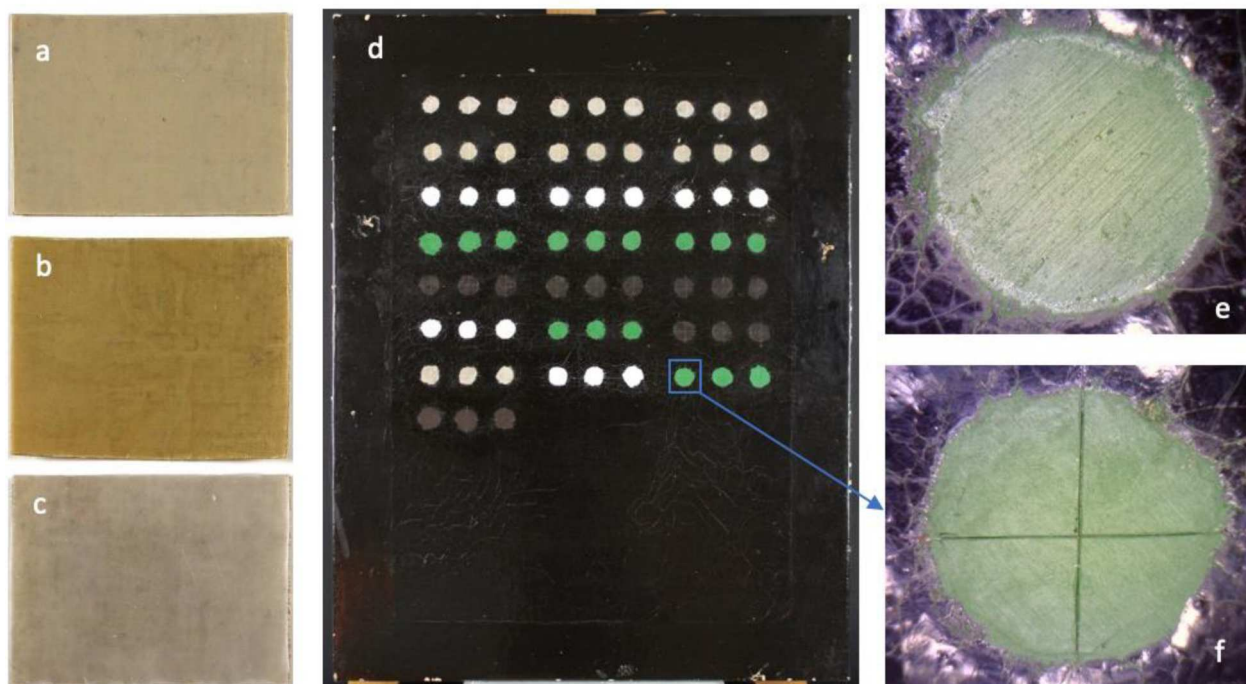


Figure 2. NOVA-PWR with chalk (a), kaolin (b) and aluminum hydroxide (c) applied on pieces of sized canvas (8 cm x 6 cm), specifically for flexibility tests; (d) Model painting (35 cm x 45 cm) with losses (1 cm in diameter) filled with PWR: losses filled with NOVA-PWR with chrome oxide green, for fluctuating RH (e) and fluctuating T (f) trials.

number of Byko Opacity 2A Test Charts. Therefore, PWR formulations were applied in a thin film onto sheets of polyester film (Melinex 100 μ) using a small heated spatula (a Whipmix Wax Carving Pencil, see Appendix 3). Although a completely uniform thickness could not be achieved, the PWRs were applied consistently by one operator. Opacity evaluation was made by visual comparisons between the different formulations; a practical evaluation which was enough to establish clear trends in the degree of opacity according to the ratio of ingredients (Figure 3).

Flexibility: Tests with different fillers in different ratios were conducted to establish which formulations could withstand bending without breaking. The “Standard Test Methods for Bend Testing of Material for Ductility” – ASTM E290-14 (ASTM 2014) calls for a mandrel or plunger of defined dimensions which were not available for this study. Instead, 11 laboratory beakers with different diameters were used for bending the surfaces. Two different ratios of WR (either 1.5 or 2 parts of Cosmoloid H80 to 1 part of Regalrez 1126, by weight) were combined with three different fillers (Champagne chalk, kaolin and aluminum hydroxide) in different amounts (Appendix 2). These were applied onto linen as thin even coatings using a Hydraulic Dry Mounting and Laminating Heat Press (see Appendix 3). The canvas was previously sized with Plextol B500. Trials were performed manually by the same person with one sample from each formulation. The results, while certainly not as objective as using the ASTM standard, were enough to provide a broad indication of behavior in relation to the different formulations and for evaluating behavior of WR with fillers in thin layers

applied over a large canvas insert (see Rocha Pires et al. 2023) (Figure 4).

3.3. Methods to evaluate stability

3.3.1. Environmental chamber and radiant heat trials

Samples from 18 NOVA-PWR formulations and those made from 4 Gamblin-PWRs (Appendix 2), were exposed to stability tests. Since a primary goal was to study bloom development in samples with beeswax versus the NOVA-PWRs, conditions in Isa von Lenthe’s research on the stability of beeswax in beeswax-chalk-pigment infill materials were used as a guide (von Lenthe 2019). Trials 1–4 were carried out in an Aralab FITOCLIMA 300 EC20 environmental chamber. The rectangular and circular samples that were subjected to the environmental trials were placed on top of a glass plate partially covered with a Reemay 2004 (see Appendix 3) acid-free polyester tissue. The Reemay was not completely attached to the glass, allowing for air to circulate under the samples. The infilled model paintings were placed horizontally in the climate chambers for trials 1 and 2. The radiant heat trial (5) aimed to establish whether PWRs would be softened by sunlight striking a painting (either through window glass or an open window).

- Trial 1: Fluctuating RH (10-90%) and stable T (25°C), for 600 h, with 12-hour cycles. To determine if any undesirable surface characteristics would result from exposure to fluctuating cycles of RH and to confirm the role of fluctuating RH in the

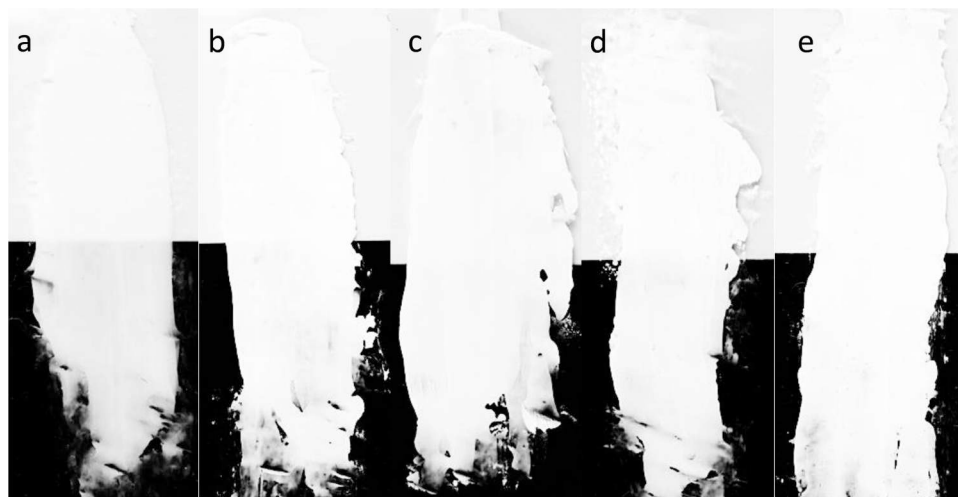


Figure 3. Opacity tests of PWR (in the ratio of 1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126, by weight), containing titanium white in 0.125 parts (a), 0.5 parts (b) and 1 part (c) to 1 part of wax-resin binder; (d) titanium white and Champagne chalk (1:1) in 1 part to 1 part of wax-resin binder; (e) Gamblin-PWR labeled “Titanium White.” Each PWR application is approximately 8 cm in length.

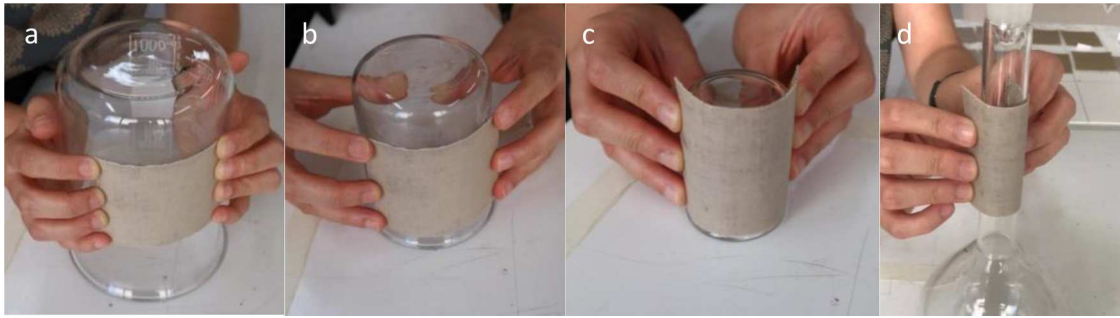


Figure 4. PWR sample in the ratio of 1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126 (by weight), with Champagne chalk (1.25 parts), during bending tests on cylinders with the following diameters: 13 cm (a), 6 cm (b), 4 cm (c) and 2 cm (d). The PWR mixture is on the outside of the bend.

development of bloom on Gamblin-PWR samples (Figure 5).

- Trial 2: Fluctuating T (10–40°C) and stable RH (55%), for 600 h, with 12-hour cycles. To establish the role of fluctuating T in relation to the stability of NOVA-PWR formulations, and whether bloom on Gamblin-PWR samples can be associated with fluctuating T. Trial 2 also aimed to study the effect of T on softening (with higher T) or shrinkage (with lower T). Softening was to be assessed mainly by observing the shape of the circular protrusion in the rectangular samples and the lines scored in the filled losses in the model paintings. Shrinkage was assessed using the test samples applied to the model paintings (Figure 6).
- Trial 3: High T of 50°C and uncontrolled RH, for 3 h in total. Trials 3 and 4 were conducted since no visible

changes due to T in Trials 1 and 2 were observed. In Trial 3, T was increased from room T to 50°C in one hour, maintaining that T for one hour, and then decreasing it to room T for one hour.

- Trial 4: High T of 60°C and uncontrolled RH, for 3 h in total. The same conditions as Trial 3 but with T increasing to 60°C (Figure 7).
- Trial 5: Exposure to radiant heat up to 70°C. The effect of sunlight was simulated by irradiating loose samples at a distance of ~3 cm from an incandescent light bulb (Philips), with 670 lumen, 60 W, and 230 volts. An infrared thermometer (see Appendix 3) was used to constantly measure the surface of the sample, while macro-photographs were taken at every 2.5°C increase. Each sample was irradiated for approximately 40 min, until the surface T reached 70°C.

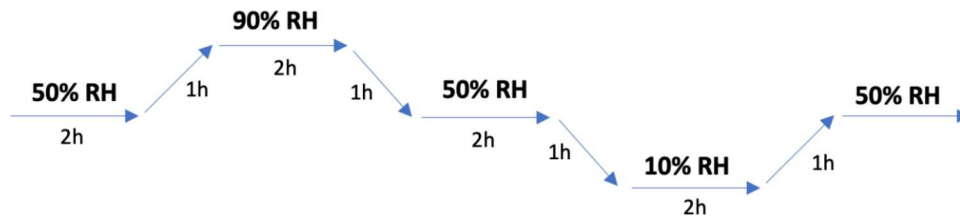


Figure 5. RH fluctuations in the first environmental trial. T was kept at 25°C.

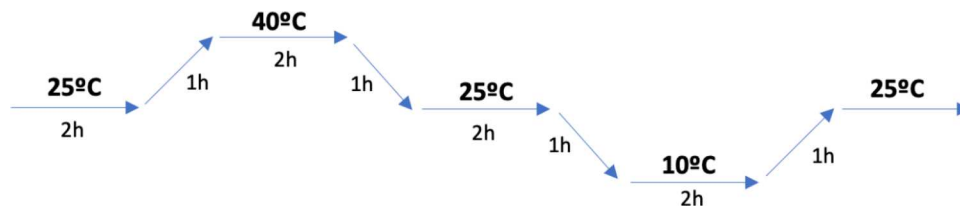


Figure 6. T fluctuations in the second environmental trial. RH was kept at 55%.

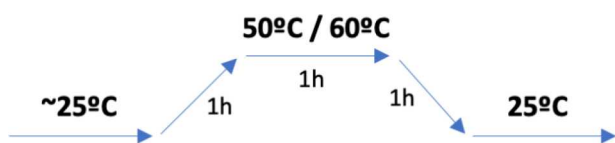


Figure 7. T values in the third (50°C) and fourth (60°C) environmental trials. RH was not controlled.

3.4. Methods to evaluate testing results

Assessments of visual and physical changes were made with macro photography, digital microscopy, hardness measurements and ATR-FTIR analysis. Macro photographs were taken before and after exposure with a Canon EOS M10 digital camera with a Canon Macro Lens EF-M 28 mm 1:3.5 IS STM which documented surface changes (bloom, blisters, or differences in gloss). A set of samples was removed halfway through each trial and photographed.

High resolution surface details were acquired with digital microscopes. A Dino-Lite Digital Microscope PRO AD413 (kindly loaned by Professor Dr. Jaap Boon) at magnifications from 20x to 50x was used to record samples before, during and after exposure in the environmental chamber and to radiant heat. To document any changes due to softening, samples with circular protrusions were held on edge in slots on a wooden stand with the digital microscope positioned above (Figure 11). A Dino-Lite Edge Optical Microscope AM7915MZTL with and without a polarization filter was used to document bloom on Gamblin-PWR samples (50x to 85x magnification). The DinoCapture 20.0 programme was used to visualize, capture and process the images.

Hardness measurements were taken with a digital Shore D durometer, before, during and after exposure.

A selection of pigmented and unpigmented NOVA-PWR and Gamblin-PWR samples were analyzed using ATR-FTIR to determine whether exposure to fluctuating RH and T, or extreme T induced detectable molecular changes. Spectra were obtained using an Agilent Handheld 4300 spectrophotometer equipped with a wire-wound source, a ZnSe beam splitter, a Michelson interferometer, and a thermoelectrically cooled DTGS detector and acquired with a diamond ATR module at 128 scans and 4 cm⁻¹ resolution, between 4000 and 650 cm⁻¹. Spectra were analyzed with Omnic 8 software but edited and normalized (to [0, 1]) in Origin Pro 2016.

3.5. The compatibility of NOVA-PWRs with varnish coatings and inpainting media

The successful NOVA-WR formulation (1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126, by weight)

was used to prepare PWRs with Champagne chalk, chrome oxide green, burnt umber, and titanium white, as well as one without pigment/filler (Appendix 2). Applied in horizontal strips over a commercially prepared canvas (see Appendix 3), samples were textured, using a silicone mold, to evaluate changes after application of the varnishes and inpainting media.

Six synthetic resin varnishes (Paraloid B-72 in 1-methoxy-2-propanol in three different dilutions: 10%, 5% and 2.5%, 10, 5 and 2.5 g in 100 ml, respectively; Paraloid B-72 10% in ethanol, 10 g in 100 ml; Laropal A81 20% in Shellsol A and Shellsol D40, 20 g in 40 ml of Shellsol A and 60 ml of Shellsol D40; and Regalrez 1094 25% in Shellsol T, 25 g in 100 ml) were applied by brush on top of the different samples, with a separate area left unvarnished. After drying, six different inpainting media (Gamblin Conservation Colors in Laropal A81, Kremer Retouching chips in Paraloid B-72, Kremer Retouching colors in Shellac, Golden Acrylics and Kremer watercolors in Gum Arabic) were applied on top on the varnish layers and over the unvarnished area.

Once dry, the inpainting media were removed using two different solvents (white spirits with ~17% aromatics and ethanol 96%) to determine whether the underlying varnish layer was sufficient to protect the PWR infills from being dissolved by solvents.













4. Results and discussion

4.1. Characterization of the NOVA-PWR in comparison with the Gamblin-PWR

The most promising results in terms of hardness and workability were obtained with either 1.5 or 2 parts of Cosmoloid H80 to 1 part of Regalrez 1126 (by weight). Of the two, the formulation with 2 parts of Cosmoloid H80 was the closest to the Gamblin-PWR “Neutral Base.” However, this ratio developed blisters when exposed to 60°C and 70°C (Table 2) whereas the ratio with 1.5 parts of Cosmoloid H80 remained stable in the same conditions and was still close to the Gamblin-PWR in workability and hardness. Detailed information in Rocha Pires (2021).

The amount of filler and pigment added to the mixture of 1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126 (by weight) can be adjusted significantly to achieve a range of properties in terms of transparency/opacity as well as hardness, without compromising the stability of the NOVA-PWR. In general, the pigments had much higher hiding power than the fillers. When added to each of the pigments, Champagne chalk lowered the

Table 2. Surface of unpigmented NOVA-WR infills in different wax-resin ratios, by weight, after exposure to T up to 60°C. The scale in the bottom right corner of all images corresponds to 0.1 cm.

1 part of Cosmoloid H80 to 2 parts of Regalrez® 1126	1 part of Cosmoloid H80 to 1.5 parts of Regalrez® 1126	1 part of Cosmoloid H80 to 1 part of Regalrez® 1126	1.5 parts of Cosmoloid H80 to 1 part of Regalrez® 1126
			
2 parts of Cosmoloid H80 to 1 part of Regalrez® 1126	2.5 parts of Cosmoloid H80 to 1 part of Regalrez® 1126	3 parts of Cosmoloid H80 to 1 part of Regalrez® 1126	3.5 parts of Cosmoloid H80 to 1 part of Regalrez® 1126
			
4 parts of Cosmoloid H80 to 1 part of Regalrez® 1126	4.5 parts of Cosmoloid H80 to 1 part of Regalrez® 1126	5 parts of Cosmoloid H80 to 1 part of Regalrez® 1126	Cosmoloid H80 100%
			

opacity of the mixture compared with pigments alone, particularly with burnt umber. These findings indicate that the transparency/opacity of the infill can be easily adjusted to suit the painting.

Flexibility tests showed that the greater the amount of filler added, the less capable of withstanding bending without breaking is the formulation. Among the fillers tested, Champagne chalk showed the best results. Mixtures with 0.5 parts of Champagne chalk to 1 part of WR binder only started to crack when bent around a cylinder with the smallest diameter (2 cm). Since the wax to resin ratio of 1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126 (by weight) was important for temperature stability, adjusting the wax content to achieve greater flexibility was not considered viable (Table 2).

4.2. Stability of the NOVA-PWR to the environmental conditions tested

Five parameters were evaluated: development of bloom, stability to high T, embrittlement and/or cracking, changes in hardness, and molecular changes evident with ATR-FTIR.

4.2.1. Development of bloom

Gamblin-PWRs developed bloom in both fluctuating RH (10–90%) with constant T (25°C) (Figure 8), and fluctuating T (10–40°C) with constant RH (55%) (Figure 9), although the resulting appearance differed and was more intense with fluctuating T. It is important to note that, while the environmental and, in particular, the heat exposure studies involved extreme conditions, the Gamblin-PWRs had already developed clearly visible bloom while stored in their original container in ambient conditions. It is also worth highlighting that previous studies reported that a coating of varnish over WR formulations with beeswax can inhibit bloom formation (von Lenthe 2019) and all the samples tested in this investigation were unvarnished.

While bloom was most clearly visible on the glossy half of Gamblin-PWR rectangular samples, it was not visible on Gamblin-PWRs applied to losses in the model painting after having been removed from the climate chambers. In the latter case, the infills were matte, as a result of being leveled with solvent. On a later examination, approximately one month after the trials, all the Gamblin-PWRs had developed bloom, of higher or lower intensity.

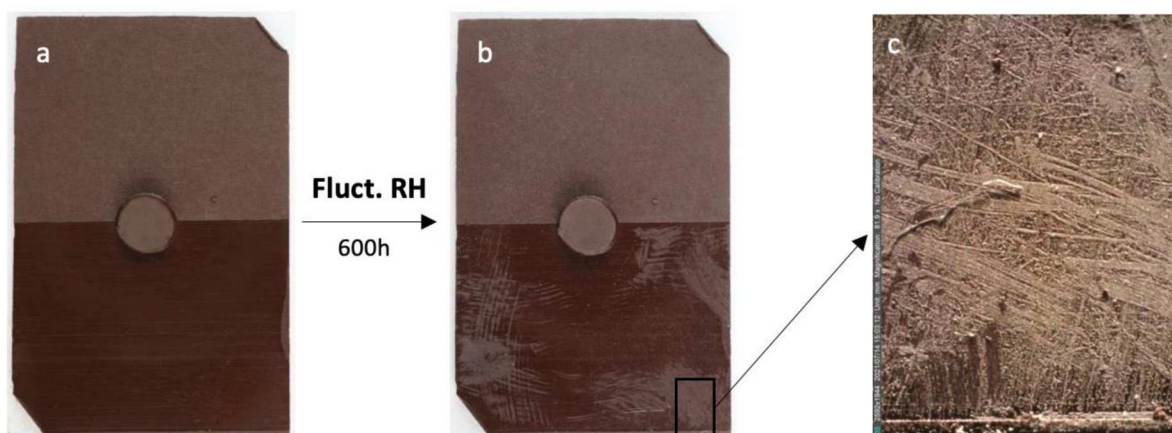


Figure 8. Gamblin-PWR labeled “Burnt umber” (3.4 cm × 2.3 cm × 0.1 cm) before (a) and after (b) being exposed to 600 h of fluctuating RH. Detail of the topography of the bloom layer taken with a digital microscope and raking light (c). Red scale in (c) corresponds to 0.05 cm.

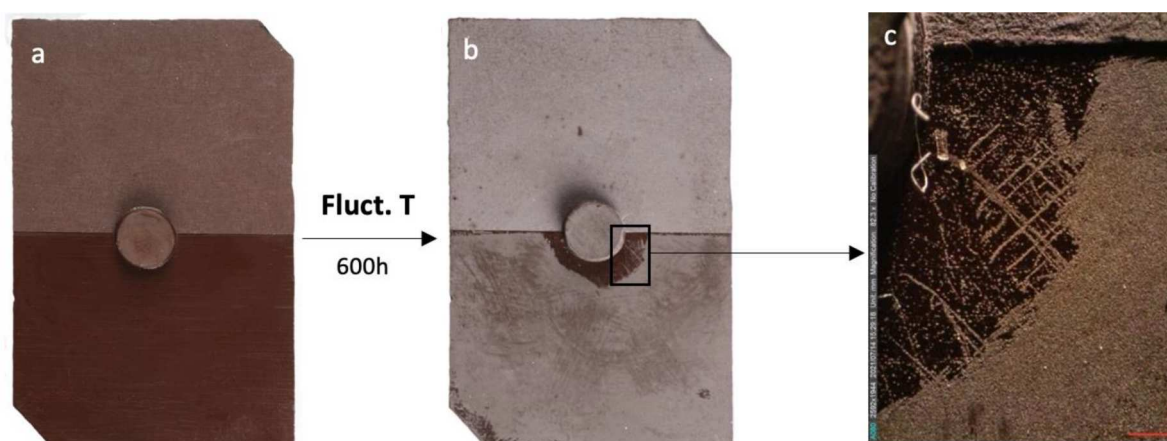


Figure 9. Gamblin-PWR labeled “Burnt umber” (3.4 cm × 2.3 cm × 0.1 cm) before (a) and after (b) being exposed to 600 h of fluctuating T. Detail of the topography of the bloom layer taken with a digital microscope and raking light (c). Red scale in (c) corresponds to 0.05 cm.

The NOVA-PWRs did not develop bloom in the same testing conditions (Figure 10). ATR-FTIR analysis of the bloom layer from a sample of Gamblin-PWR “Burnt UMBER” subjected to fluctuating RH was very similar to a beeswax reference sample (Rocha Pires 2021), confirming previous studies suggesting that bloom results from a migration to the surface of components in beeswax.

4.2.2. Stability to high T (40°C, 50°C, 60°C) and radiant heat (up to 70°C)

The most significant result was the development of blisters at the surface of unpigmented NOVA-WR samples with high ratios of Cosmoloid H80 wax (from 2 parts of Cosmoloid H80 by weight, upwards), after being exposed to a T of 60°C (Table 2). These results are from a pilot environmental trial (which

had a maximum T of 60°C), which is not explained in detail in this paper for having been unsuccessful for the desired outcomes. It did, however, give insight into the stability to T up to 60°C of unpigmented NOVA-WR mixtures in different ratios of wax to resin. The Gamblin-PWR were not tested at this point.

While it is highly unlikely that in normal circumstances a painting would be exposed to such an extreme T, it was felt that the stability displayed by the ratio of 1.5 parts Cosmoloid H80 provided a high level of confidence for T stability. The NOVA-WR formulation recommended for use by conservators is, therefore, 1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126 (by weight).

None of the samples (NOVA-PWR, made with the ratios of 1, 1.5 or 2 parts of Cosmoloid H80 to 1 part

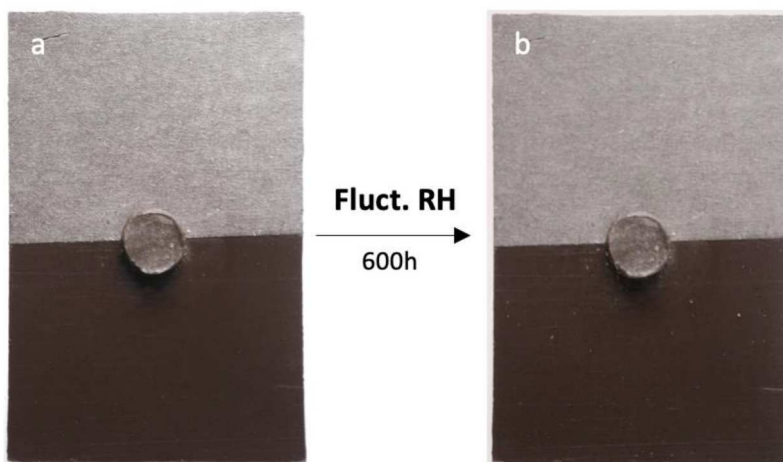


Figure 10. NOVA-PWR sample (3.4 cm × 2.3 cm × 0.1 cm) in the ratio of 1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126 (by weight), with burnt umber (0.75 parts), before (a) and after (b) being exposed to 600 h of fluctuating RH. The top half of the sample has a matte surface, whereas the bottom half is glossy.

of Regalrez 1126 by weight, and Gamblin-PWR), subjected to fluctuating T up to 40°C and to fixed Ts of 50°C and 60°C showed any visible changes due to softening. However, some free-standing samples subjected to radiant heat (up to 70°C) showed a slight overall curvature when observed from the side (Figure 11). This is not expected to happen if the PWR infills are introduced in a loss with a support present below.

4.2.3. Cracking

Cracking was observed in losses in the model paintings exposed in trials 1 and 2 in the environmental chamber (Figure 12). Materials present in the model paintings may have contributed since the “paint” layers consisted of pigmented dammar resin, not oil paint. Moreover, these model paintings had already undergone substantial mechanical cracking prior to having losses created and infilled for this study (Reis 2011).

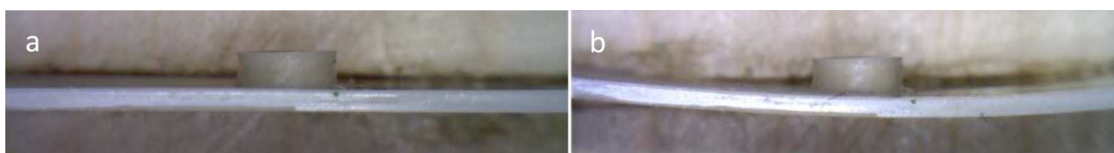


Figure 11. Side view of a NOVA-PWR sample in the ratio of 1.5 parts of Cosmoloid to 1 part of Regalrez 1126 (by weight) with Champagne chalk (1.25 parts), before (a) and after being exposed with radiant heat (up to 70°C). The circular protrusion in the center has a thickness of approximately 0.2 cm.

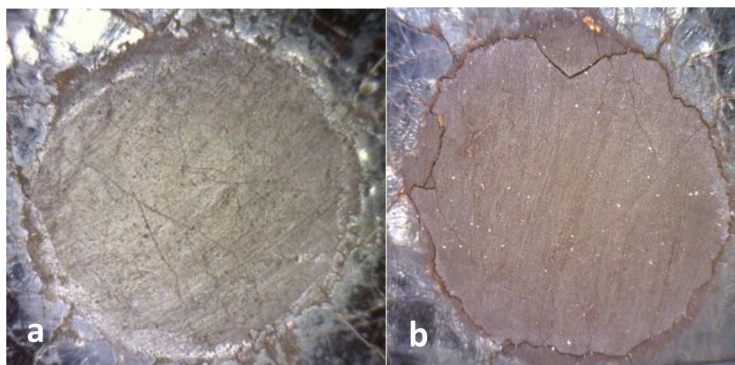


Figure 12. Filled losses (1 cm in diameter) in the model painting after 600 h of fluctuating RH: (a) NOVA-PWR in the ratio of 1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126 with burnt umber (0.75 parts); (b) Gamblin-PWR labeled “Burnt UMBER.”

The differences in location and appearance of cracks in the NOVA-PWRs and Gamblin-PWRs are interesting: while the NOVA-PWR cracks cross over the surface and align with cracks in the model painting, the interface between the model paint and infill remains secure (Figure 12a). In contrast, the Gamblin-PWR infills are not cracked across, but show a circular crack line apparently at the interface with the model paint (Figure 12b). In the latter case, the circular cracking indicates failure at the edges of the loss, perhaps indicating a lack of adhesion, and/or significant resistance to dimensional changes in the canvas/“paint” composite. The difference in the location and type of mechanical failure in the PWRs requires further research to determine how the cracking relates to differences in their physical properties and in what precise conditions the cracking occurs.

4.2.4. Changes in hardness

While hardness measurements were not conclusive because the standard deviation was higher or very close to the calculated variations, results suggest that an increase in hardness may be related to fluctuating RH and T (possibly to lower T) and that a decrease in hardness may have resulted from exposure to higher T. Details on hardness results in Rocha Pires (2021). Further research on the effect of different environmental conditions on the hardness of the PWRs is required.

4.2.5. Chemical changes visible with ATR-FTIR

No visible differences in the ATR-FTIR spectra of the NOVA-PWR samples were detected before and after environmental trials (Figure 13a). However, a new band at 1126 cm^{-1} (for which no attribution was found in the literature) was detected in the Gamblin-PWR spectra from pigmented and unpigmented

samples after exposure (Figure 13b). This and other minor changes in the ATR-FTIR spectra for the Gamblin-PWR samples are not sufficient for evidence of deterioration. They may be related to physical changes and the movement of components within the samples which cause different bands to become more visible or intense. Further research is required.

4.2.6. Compatibility with varnishes and inpainting media

Results showed that a varnish coating of Paraloid B-72 in 1-methoxy-2-propanol can be applied on top of the NOVA-PWRs without dissolving them. It is important to note, however, that 1-methoxy-2-propanol, for its “high-moderate” swelling action on oil paint (Phenix and Wolbers 2021, 572) should only be used in a local isolation layer over the fills and not as solvent for varnishing oil paintings.

A coating of 10% Paraloid B-72 varnish offered the most effective protection from solvents such as white spirit (~17% aromatics) which could be used to remove upper layers of inpainting materials. Ethanol (96%) had almost no effect on the infills. Widely available inpainting media, such as Gamblin Conservation Colours in Laropal A81, were found to be compatible with both the varnished and unvarnished areas. Further details in Rocha Pires 2021.

5. Conclusions and recommendations

A WR formulation consisting of 1.5 parts of Cosmoloid H80 microcrystalline wax to 1 part of Regalrez 1126 synthetic resin (by weight), used either unpigmented or mixed with pigments and/or fillers, has proven to be an effective and stable alternative to PWR infills containing beeswax. This PWR formulation is of particular

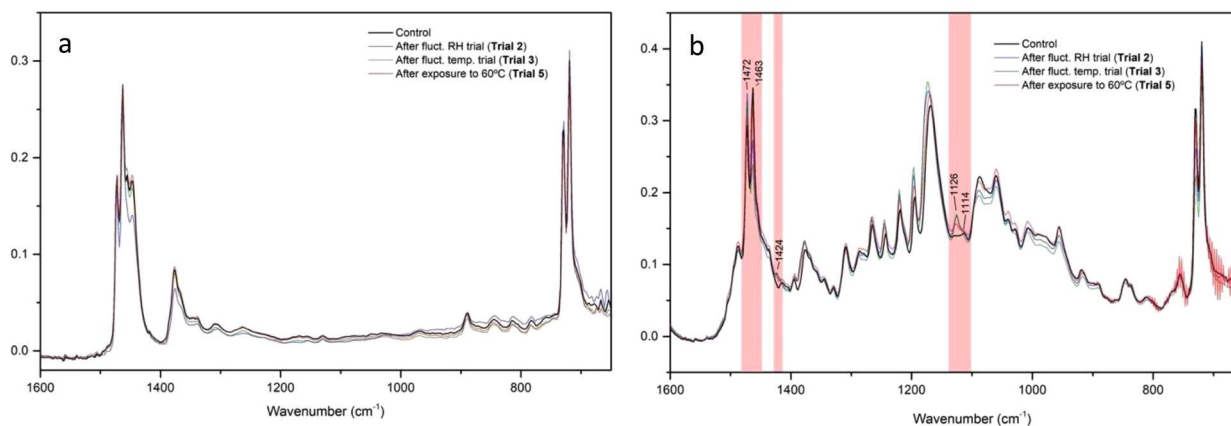


Figure 13. Overlapped ATR-FTIR spectra of an unpigmented NOVA-WR with 1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126 (a); and an unpigmented Gamblin-WR labeled “Neutral Base” (b), with spectral differences in red. These are details of the spectra, in the region of $1600\text{--}600\text{ cm}^{-1}$ where spectral differences were observed.

value for infilling paintings which will not be inpainted or varnished, and for infilling losses on paintings on copper (where the acidic nature of beeswax could have a corrosive effect on the support). The quantities used for all ingredients (including pigments and fillers) are given in the Appendix 2.

This research made use of a combination of sophisticated instrumentation, such as ATR-FTIR, with more practical approaches and tests. All provided useful and complementary data, allowing for a holistic understanding of the behavior and stability of the PWR formulations.

Results confirmed that a WR formulation with beeswax can develop visually disturbing bloom composed of beeswax components when subjected to fluctuations in RH and T. Formulations with Cosmoloid H80 in combination with Regalrez 1126 resin did not develop bloom in the same conditions.

A key finding is that the ratio of wax to resin in the NOVA-PWR formulation is most stable to high T when kept within the range of 1.5 parts of Cosmoloid H80 to 1 part of Regalrez 1126 (by weight).

While exact color matching is easily achieved for losses in paint with a uniform color (or in small losses), results showed that it is also possible to use the NOVA-PWR as a base to accept layers of inpainting media. The best results were found with a varnish layer of 5 or 10% Paraloid B-72, although it is possible to employ other synthetic coating resins, followed by inpainting with Gamblin Conservation Colors (in Laropal A81). Once sealed with Paraloid B-72 either directly over the infills alone or with inpainting on top, the NOVA-PWR infills can accept brush or spray coats of other varnishes normally used by paintings conservators.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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Appendix

Appendix 1: Overview of wax and wax-resin recipes used for infilling and reintegrating losses

Author/ Institution	Year	Wax(es)	Resin(s)	Other material(s)	Proportion of materials	Purpose	Application	Reference
Perney	1757	Wax (type not mentioned)	Pitch of resin (type not mentioned)	Bricks pounded and sifted	Not mentioned	Infilling	Not mentioned	Pemey 1757, 406
Fratel	1770	Wax (type not mentioned)	Not included	Oil paint	Wax and oil paint in equal proportions	Infill and reintegrate oil paint losses	With a palette knife	Ollendorf 1999; von Lenthe 2019, 9
Prange	1828	Wax (type not mentioned)	Not included	Not mentioned	Not mentioned	Infilling disturbing cracks	Not mentioned	Wagner 1988; von Lenthe 2019, 9
Lucanus	1829	Wax (type not mentioned)	Not included	Chalk/chalk paste	Not mentioned	Infilling	Everything dissolved in turpentine	Lucanus 1929, 62; von Lenthe 2019, 9
De Montabert	1834	Melted wax (type not mentioned)	Not included	Volatile oil	Not mentioned	Infilling	Wax applied with a warm knife and smoothed with a polishing stone	Paetzold 1994, 47; von Lenthe 2019, 9
Hampel	1846	Fresh yellow wax	Not included	Linseed oil, brown chalk, finely ground chalk or lead white	2:1:1:1 (wax:oil:brown chalk:white chalk)	Infilling	Not mentioned	Hampel 1846, 29; von Lenthe 2019, 10
Déon	1851	Virgin natural wax	Not included	Not mentioned	Not mentioned	Infilling losses in panel paintings	Not mentioned	Déon 1851, 20–21
Prager	1927	Wax (type not mentioned)	Resin (type not mentioned)	Not mentioned	Not mentioned	Infilling losses in panel paintings	Not mentioned	Paetzold 1994, 48; von Lenthe 2019, 10
Kudrjawzew	1954	Wax (type not mentioned)	Resin (type not mentioned)	Dry pigments	Not mentioned	Infilling and retouching	Different colored wax-resins mixed together to form varying color tones	Kudrjawzew 1954, 137; von Lenthe 2019, 10
Brachert	1955	Melted wax (type not mentioned)	Not included	Stand oil and Champagne chalk	Not mentioned	Infilling	Not mentioned	Brachert, 1955; von Lenthe 2019, 10
Althöfer	1959	Polyethylene glycols	Not included	Chalk, pigments, beeswax	3:2:3 (Polywax 12000: chalk)	Infilling and retouching	Not mentioned	Althöfer 1959, 33
Ruhemann	1968	Beeswax or relining wax	Not included	Gilder's whitening or pigment, drying oil, secotone glue and zinc white	Not mentioned	Infilling losses in easel paintings	Mixture executed on a warm surface and pressed into the losses with a small metal modeling spatula or scalpel; excess removed with silk humidified in water or turpentine; smoothing the surface with talcum powder	Ruhemann 1968, 241

(Continued)



Continued.

Author/ Institution	Year	Wax(es)	Resin(s)	Other material(s)	Proportion of materials	Purpose	Application	Reference
Kelly	1973	Beeswax	Not included	Insect repellent	Not mentioned	Infilling wormholes in wood	Wax applied with a heated spatula	Kelly 1979, 194; von Lenthe 2019, 10
Emile-Måle	1976	Wax (type not mentioned)	Not included	Chalk; pigments	Not mentioned	Infilling and retouching modern pictures	With a heated spatula and pallet	Emile-Måle 1976, 88
Canadian Conservation Institute	1980s	Beeswax	Ketone Resin N	Pigment; chalk	4:1 (wax:resin)	Infilling and retouching losses in paintings	Not found	Vega 2016, 25; Fuster-López 2021, 618
Schneider	1981	Beeswax	Colophony and Alkidal (synthetic resin 50% + linseed oil 50%)	Methylcellulose; zinc white with glue + French chalk; English whitening, zinc white; pigment (if required)	1:1:1:2 (beeswax and colophony; alkidal; Methylcellulose: zinc white with glue) + 1:1:1 (chalk:whitening; zinc white)	Infilling losses in oil paintings on canvas	Applied in thin layer with a flexible spatula	Schneider 1981, 44
Schneider	1981	Beeswax	Colophony and Alkidal	Methylcellulose; Japanese paper (shredded); 10% solution of sturgeon bladder	1:1:2:4 (beeswax and colophony; alkidal; methylcellulose; Japanese paper)	Infilling losses in paintings on cardboard	Applied in thin layer with a flexible spatula	Schneider 1981, 45
Leegenhoek-Wade	1983	Beeswax	Not included	Calcium carbonate	Not mentioned	Infilling losses in paintings on copper	Not mentioned/not found	Leegenhoek 1983; Vega 2016, 22
Althöfer	1985	Beeswax	Dammar resin	Champaign chalk	3:2:1 (wax:resin:chalk)	Infilling losses in contemporary paintings	With a thermic spatula and a pre-heated dentist spatula and smoothened with a swab soaked in white spirit	Althöfer 1985, 108
SRAL	1990-today	Paraffin wax (in BEVA 371/ BEVA 371b)	Ketone resin (in BEVA 371)/ aldehyde resin (in BEVA 371b)	Kaolin/Portafill 40/chalk; polymer fraction (EVA), plasticizer, solvent (in BEVA 371/ BEVA 371b)	Varying proportions depending on the purpose of the material	Infilling and retouching losses in paintings	To apply the filler a small metal dental tool can be heated and pressed over the silicon coated Melinex on top of the filler	K. Seymour (pers. comm. 2019); Brites, Carlyle, and Marques 2015, 75
Garrel	1992	Beeswax	Not included	Not found	Not found	Infilling losses in paintings on copper	Not found	Garrel 1992, Vega 2016, 22
Gänsicke and Hirx	1997	Ethylene acrylic acid copolymers A-C 540 and 580	Polyvinyl Acetate AYAC	Antioxidants Irganox 1076 or 1035; fillers	Not mentioned	Infilling losses in translucent materials	Not mentioned	Gänsicke and Hirx 1997, 24
Nicolaus	1999	Beeswax	Not included	Champagne chalk or Bolognese chalk	Not mentioned	Infilling losses in paintings	Mixture prepared on a metal plate and applied with a spatula	Nicolaus 1999, 241
		Beeswax	Not included	Pigment	Not mentioned			
		Beeswax	Dammar resin	Champagne chalk or Bolognese chalk	3:1 (wax:resin)			
Kemp	2009	Beeswax Microcrystalline wax	Colophony	Venice turpentine; chalk	3:1:1 (wax:resin: turpentine)	Infilling large-crystal translucent marbles	Not mentioned	Kemp 2009, 70

(Continued)

Continued.

Author/ Institution	Year	Wax(es)	Resin(s) Resin (type not mentioned)	Other material(s) Dry pigments	Proportion of materials Not mentioned	Purpose Infilling losses in marble	Application Not mentioned	Reference Kemp 2009, 74
Folkes and Reddington	2010	Beeswax	Dammar resin	Chalk	3:1 (wax:resin)	Infilling complex surfaces	Applied with a thermostatically controlled heated spatula; cotton wool swabs with white spirit to remove excess; texturing done with previously prepared silicone molds	Folkes and Reddington 2010, 159–161
Buffalo State College (NY)	Before 2011	Beeswax + BeSquare 195	Laropal K-80	Pigments	3:1:1:5 (Beeswax: BeSquare™195: Laropal K-80)	Infilling	Not found	McIntyre 2011
McIntyre	2011	Beeswax + BeSquare 195	Laropal A-81	Pigments	3:1:1:5 (Beeswax: BeSquare 195: Laropal A-81)	Infilling and retouching	Wax and resin mixed in a double boiler on a hot plate; applied with spatulas	McIntyre 2011, 20
Gamblin	2014	Beeswax + BeSquare 195	Laropal A-81	Pigments	3:1:1:5 (Beeswax: BeSquare 195: Laropal A-81)	Infilling and retouching	Applied with a wax-carving pencil	Gamblin Conservation Colours n.d.; McIntyre 2011 Vega 2016
Vega	2016	Techniwax 9426	Regalrez 1094	Pigment + chalk	4:1 (wax:resin)	Infilling and retouching	Wax and resin mixed in a double boiler on a hot plate; applied using a 'wax carving pencil'	González 2017
González	2018	Cosmoloid H80	Regalrez 1126	Pigment + chalk	1.5:1 (wax:resin)	Infilling and retouching	Wax and resin mixed in <i>bain marie</i> in a hot plate	von Lenthe 2019, 2
Von Lenthe	2019	Beeswax and Cosmoloid H80	Not included	Chalk and pigment	1:3:2.5 (beeswax: Cosmoloid H80:chalk)	Infilling and retouching	Prepared in a hot plate and applied with heated spatulas	von Lenthe 2019, 2

Appendix 2: NOVA-PWR formulations used in the research

Successful formulations in workability and stability are highlighted in green; successful formulations in workability, but not tested for stability, are highlighted in blue.

		Formulations			Tested for (*)
	Cosmoloid H80 (parts by weight)	Regalrez 1126 (parts by weight)	Filler/Pigment (parts by weight)		
Unpigmented	1 part (3.3g)	2 parts (6.7g)			W, H, S (**)
	1 part (4g)	1.5 parts (6g)			W, H, S (**)
	1 part (5g)	1 part (5g)			W, H, S
	1.5 parts (6g)	1 part (4g)			W, H, S
	2 parts (6.7g)	1 part (3.3g)			W, H, S
	2.5 parts (7.1g)	1 part (2.9g)			W, H, S (**)
	3 parts (7.5g)	1 part (2.5g)			W, H, S (**)
	3.5 parts (7.8g)	1 part (2.2g)			W, H, S (**)
	4 parts (8g)	1 part (2g)			W, H, S (**)
	4.5 parts (8.2g)	1 part (1.8g)			W, H, S (**)
5 parts (8.3g)	1 part (1.7g)			W, H, S (**)	
With fillers	1 part (5g)	1 part (5g)	1 part <u>Champagne chalk</u> (5g)		W, H, S
	1.5 parts (6g)	1 part (4g)	1.25 parts <u>Champagne chalk</u> (5g)		W, H, O, F, S
	1.5 parts (6g)	1 part (4g)	2.5 parts <u>Champagne chalk</u> (10g)		W, H, O, F
	1.5 parts (6g)	1 part (4g)	5 parts <u>Champagne chalk</u> (20g)		W, H, O, F
	2 parts (6.7g)	1 part (3.3g)	1.5 parts <u>Champagne chalk</u> (5g)		W, H, F, S
	2 parts (6.7g)	1 part (3.3g)	3 parts <u>Champagne chalk</u> (10g)		W, H, F
	2 parts (6.7g)	1 part (3.3g)	6 parts <u>Champagne chalk</u> (20g)		W, H, F
	1.5 parts (6g)	1 part (4g)	1.25 parts <u>kaolin</u> (5g)		W, H, O, F
	1.5 parts (6g)	1 part (4g)	2.5 parts <u>kaolin</u> (10g)		W, H, O, F
	1.5 parts (6g)	1 part (4g)	5 parts <u>kaolin</u> (20g)		W, H, O, F
	2 parts (6.7g)	1 part (3.3g)	1.5 parts <u>kaolin</u> (5g)		W, H, F
	2 parts (6.7g)	1 part (3.3g)	3 parts <u>kaolin</u> (10g)		W, H, F
	2 parts (6.7g)	1 part (3.3g)	6 parts <u>kaolin</u> (20g)		W, H, F
	1.5 parts (6g)	1 part (4g)	1.25 parts <u>aluminium hydroxide</u> (5g)		W, H, O, F
	1.5 parts (6g)	1 part (4g)	2.5 parts <u>aluminium hydroxide</u> (10g)		W, H, O, F
	1.5 parts (6g)	1 part (4g)	5 parts <u>aluminium hydroxide</u> (20g)		W, H, O, F
	2 parts (6.7g)	1 part (3.3g)	1.5 parts <u>aluminium hydroxide</u> (5g)		W, H, F
	2 parts (6.7g)	1 part (3.3g)	3 parts <u>aluminium hydroxide</u> (10g)		W, H, F
	2 parts (6.7g)	1 part (3.3g)	6 parts <u>aluminium hydroxide</u> (20g)		W, H, F
	With pigments	1 part (5g)	1 part (5g)	1 part <u>chrome oxide green</u> (5g)	
1.5 parts (6g)		1 part (4g)	0.3 parts <u>chrome oxide green</u> (1.25g)		W, O
1.5 parts (6g)		1 part (4g)	0.6 parts <u>chrome oxide green</u> (2.5g)		W, O
1.5 parts (6g)		1 part (4g)	1.25 parts <u>chrome oxide green</u> (5g)		W, H, O, S
1.5 parts (6g)		1 part (4g)	2.5 parts <u>chrome oxide green</u> (10g)		W, O

Formulations			Tested for (*)	
Cosmoloid H80 (parts by weight)	Regalrez 1126 (parts by weight)	Filler/Pigment (parts by weight)		
2 parts (6.7 g)	1 part (3.3g)	1.5 parts <u>chrome oxide green</u> (5g)	W, H, O, S	
1 part (5g)	1 part (5g)	0.6 parts <u>burnt umber</u> (3g)	W, H, O, S	
1.5 parts (6g)	1 part (4g)	0.3 parts <u>burnt umber</u> (1.25g)	W, O	
1.5 parts (6g)	1 part (4g)	0.6 parts <u>burnt umber</u> (2.5g)	W, O	
1.5 parts (6g)	1 part (4g)	0.75 parts <u>burnt umber</u> (3g)	W, H, O, S	
1.5 parts (6g)	1 part (4g)	2.5 parts <u>burnt umber</u> (10g)	W, O	
2 parts (6.7g)	1 part (3.3g)	0.9 parts <u>burnt umber</u> (3g)	W, H, O, S	
1 part (5g)	1 part (5g)	1 part <u>titanium white</u> (5g)	W, H, O, S	
1.5 parts (6g)	1 part (4g)	0.3 parts <u>titanium white</u> (1.25g)	W, O	
1.5 parts (6g)	1 part (4g)	0.6 parts <u>titanium white</u> (2.5g)	W, O	
1.5 parts (6g)	1 part (4g)	1.25 parts <u>titanium white</u> (5g)	W, H, O, S	
1.5 parts (6g)	1 part (4g)	2.5 parts <u>titanium white</u> (10g)	W, O	
2 parts (6.7g)	1 part (3.3g)	1.5 parts <u>titanium white</u> (5g)	W, H, O, S	
With filler + pigment	1.5 parts (6g)	1 part (4g)	0.625 parts <u>Champagne chalk</u> (2.5g) + 0.625 parts <u>chrome oxide green</u> (2.5g)	W, H, O, S
	1.5 parts (6g)	1 part (4g)	0.625 parts <u>Champagne chalk</u> (2.5g) + 0.625 parts <u>burnt umber</u> (2.5g)	W, H, O, S
	1.5 parts (6g)	1 part (4g)	0.625 parts <u>Champagne chalk</u> (2.5g) + 0.625 parts <u>titanium white</u> (2.5g)	W, H, O, S

*W: workability; H: hardness; O: opacity; F: flexibility; S: stability to environmental trials and radiant heat

**Only tested for stability to temperatures up to 60°C during the pilot environmental trial

Appendix 3: Sources of equipment and materials

Equipment and materials	Source
1-methoxy-2-propanol ≥99.5%	Sigma-Aldrich www.sigmaaldrich.com
96% ethanol	BYK Instruments www.byk-instruments.com
Byko opacity charts, BYK-Gardner GmbH, Lausitzer Str. 8, D-82538, Prufkarte 2854	Kamera Express https://www.kamera-express.nl/
Canon EOS M10 digital camera with a Canon Macro Lens EF-M 28 mm 1:3.5 IS STM	Kremer Pigmente GmbH & Co. KG www.kremer-pigmente.de
Champagne chalk (#58000)	Fruugo www.fruugo.nl
Digital infrared thermometer, Habor	Ohaus https://eu-en.ohaus.com/en-eu/
Digital precision balance standard	Fruugo www.fruugo.nl
Digital Shore D durometer hardness tester, with a sharp cone point SR0.1 mm, 30° included angle, range 0~100HD, resolution 0,5HD. Model nr.: 0226064	Available at the Department of Conservation and Restoration of FCT-NOVA
Dino-Lite Digital Microscope PRO AD413	Kindly loaned by Professor Dr Jaap Boon
Dino-Lite Edge Optical Microscope AM7915MZTL	Available in the Civil Engineering Department of FCT-NOVA
Environmental chamber Aralab FITOCLIMA 300 EC20	Gamblin Conservation Colours www.gamblincolors.com
Gamblin Conservation Colors: "Ultramarine blue," "Burnt umber" and "Indian yellow"	
Gamblin Pigmented Wax/Resin: "Neutral Base," "Burnt umber," "Chrome oxide green" and "Titanium white"	
GEPE Diapositive frames, 34 × 23 × 1 mm GP2 004	Kamera Express https://www.kamera-express.nl/
Golden Acrylics "Ultramarine blue"	Kremer Pigmente GmbH & Co. KG www.kremer-pigmente.de

(Continued)

Continued.

Equipment and materials	Source
High Intensity Daylight Lamps, article nr. 5521010, Rekoma	Deffner & Johann https://deffner-johann.de/de/
Hollow Round Hole Punch, 10 mm, REF 83270812, KWB	Leroy Merlin https://www.leroymerlin.pt/
Hot plate magnetic stirrer, 34532, Snijders	Gemini Labware b.v. www.geminibv.com
Hydraulic Dry Mounting and Laminating Heat Press on Stand, Ademco, Model 2226 IGI Wax Cosmoloid H80 microcrystalline wax	Available at SRAL The International Group, Inc. https://igiwax.com/
Inart canvas on MDF board, 50 × 60 cm, 79P5060	Ponto das Artes https://www.pontodasartes.com/
Incandescent Light Bulb, 670 lumen, 60W, 230 volts, Philips	Leroy Merlin https://www.leroymerlin.pt/
Kaolin (#58250) Kremer retouching chips in Paraloid B-72 "Ultramarine blue" Kremer retouching colors in Shellac "Ultramarine blue" Kremer watercolors in Gum Arabic "Ultramarine blue" Laropal A81 Melinex 100µ and silicone coated Melinex sheet	Kremer Pigmente GmbH & Co. KG www.kremer-pigmente.de
Paraloid B-72	Deffner & Johann https://deffner-johann.de/de/
Plextol B500	Kremer Pigmente GmbH & Co. KG www.kremer-pigmente.de
Portafill A40 (aluminum hydroxide)	CTS www.ctseurope.com/gb/
Reemay 2004 fabric	Laboratory Jan de Poorter www.ankerpoort.com
Regalrez 1094, Regalrez 1126	Restaurar e Conservar, Lda. www.restaurarconservar.com
Saga Silicone Coated Baking Paper	Kremer Pigmente GmbH & Co. KG www.kremer-pigmente.de
Shellsol A, Shellsol D40, Shellsol T	Sligro. https://www.sligro.nl/
Silicone mold C20	CTS www.ctseurope.com/gb/
Tacking Spatula Iron Thermocontrolled	Silicones & More https://www.siliconesandmore.com/nl/
Whipmix Wax CarvingPencil: Wax carving pencil	Restaurar & Conservar, Lda. www.restaurarconservar.com
White spirit EC-No. 919-446-0	https://www.gamblicolors.com/conservation-colors/ Valente e Ribeiro, Lda. www.valenteribeiro.com