

The Influence of Loading, Temperature and Relative Humidity on Adhesives for Canvas Lining

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March 12, 2020

The influence of loading, temperature and relative humidity on adhesives for canvas lining

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Abstract. The structural conservation of canvas paintings may require lining, a process in which a secondary canvas is adhered to the reverse of the damaged original canvas to provide additional support. Choosing the optimum adhesive or canvas for lining is challenging. Comprehensive data on thermal and mechanical behaviour of different adhesives to enable the conservator to make informed choices for their treatment purposes is scarce. Hence, in this study, four prevalently used adhesives for lining are chosen and their thermal and mechanical behaviour, such as the glass transition and melting temperatures, static lap shear strength and creep resistance, are compared. Thermal properties of the different adhesives are characterised using differential scanning calorimetry (DSC). Furthermore, the effect of temperature cycles (25, 35, and 45°C at a fixed relative humidity of 48%) on the creep behaviour of lined canvases is evaluated. Lap shear and creep experiments are performed on lined canvas mock-ups. The four adhesives tested are: studio formulations of an animal glue-wheat flour paste, as well as a beeswaxdamar resin mixture; a patented formula based on an ethylene vinyl acetate copolymer mixture (BEVA 371 0.F.™); and a mixture of two industrially produced acrylic copolymers (Plextol[™] D541 and K360). The results demonstrate the remarkable effect of temperature on the creep behaviour of lined canvases, which can be related to their thermal stability.

1. Introduction

A large variety of adhesive products are generally available for conservation of paintings on canvas. These can be natural or synthetic, studio formulated, engineered for the field, or commercially /industrially produced. These adhesives used for conservation/restoration purposes are mainly water or solvent-based materials. There are certain ethical stipulations for using adhesives or consolidants in conservation/restoration practice, such as being reversible, non-damaging, durable and stable in time. [1] Specifications for adhesives used for structural conservation issues were described by Berger and Zeliger in 1984, and have been considered since then as best practice [2]. However, the selection of such largely depends on the availability of the adhesive and the skills of the practitioner. In general, conservators use these adhesives based on their experience in an empirical manner. Such is the case for lining structurally impaired paintings on canvas. It is, therefore, essential that the conservation field develop a comprehensive understanding of the functional properties of these adhesives and their interaction with canvas paintings to be able to make informed choices when utilising them in conservation processes. The intrinsic stability and service life of the adhesive plays an essential role in life-time behavioural prediction of the treated artwork which is exposed to mechanical loading and environmental changes (e.g. temperature and humidity) over a long period of time. However, comparable durability data on adhesives, especially in relation to their interaction with their environment such as temperature (T) and relative humidity (RH), is still lacking.

This paper focuses on the characterisation of prevalently used adhesive mixtures for the lining of paintings. This process aims to strengthen, flatten, or consolidate oil, acrylic or tempera paintings on canvas by attaching a new canvas to the reverse of the existing one. [3] The four adhesives tested are: studio formulations of an animal glue-wheat flour paste (GP), as well as a beeswax-damar resin mixture (WR); [4] a patented formula based on an ethylene vinyl acetate (EVA) copolymer mixture (BEVA 371 O.F.TM); and a mixture of two industrially produced acrylic copolymers (PlextolTM D541 and K360). The purpose of this paper is to evaluate the thermal and mechanical performance of these four different

adhesive formulations using the same instrumentation and conditions. The long-term performance is assessed by creep testing as a function of temperature and relative humidity. Additionally, the static lap shear strength of mock-up lined samples was evaluated. The glass transition and melting temperatures of brush-outs of the adhesive samples were determined. The study focuses on lining adhesive formulations and methodologies based on those used in standard practice.

1.1 The Panorama Mesdag as a starting point of this study A nice example of a lined canvas circular painting where the mechanical creep performance of the adhesive was studied is the 'Panorama Mesdag' located in The Hague, The Netherlands. This painting gives an impression of the dunes, the sea, The Hague, and the small fishing village of Scheveningen at the end of the 19th century. It was painted around 1880 by Hendrik Willem Mesdag of the Haagse School and his team (figure 1a). It is perhaps one of the largest canvas paintings in existence, measuring some 14 metres high and 120 metres in circumference. However, due to the poor state of the exhibition facilities and, after having been exposed for over 100 years, the painting was affected by light, moisture, temperature and its own weight, combined with the effect of a leaking roof and heavy rains in 1983. This ultimately called for a largescale restoration project, including the lining of the original painting. The conservation of this enormous painting consisted of lining the complete canvas with a polyamide (Nylon 6) supporting structural fabric, bonded to the original canvas by the use of flocked BEVA 371 hot melt adhesive (figure 1b). Computer calculations and practical tests (figure 1b) were done by the Delft University of Technology (TU Delft) in The Netherlands to check the stress state and shape of the canvas. Horizontal strips of the Nylon fabric were applied successively like wallpaper, adhering each strip in sections in a continuing circular sequence.

However, some thirty years onwards, the question arose again as to how much effect the museum environment might have on the long term mechanical performance of the lining adhesive. In order to be able to answer this question, a number of lined mock-up paintings were tested with four often used lining adhesives and canvases. The results are reported in this paper. The glass transition temperatures of the adhesives were determined, as well as their initial mechanical performance, after which creep tests were performed. Focussed at museum conditions, the found results will help conservators to improve their choice for adhesive lining materials and canvases, and to assess if the 1990-1993 Panorama Mesdag lining is performing well.



Figure1: (a) Overview photograph of the circular Panorama Mesdag painting, showing the painting, the natural lighting (from top) and the central visitor platform.



Figure 1. (b) Film still of the on-scale test setup to work out the relining process in the aircraft hall of the faculty of Aerospace Engineering of TU Delft in the beginning of 1990. NPO Closeup 1996. <u>https://www.npostart.nl/close-up/11-07-2015/AT_2037854</u>

2. Materials

2.1. Canvas

The chosen lining textiles are three different fabrics. Two traditional linen canvases were used: one with closed weave (Libeco Linen 5653 ECRU, 100% linen, (25 x 25 weft x warp)) and one with a more open weave (Libeco Linen P165 ECRU, 100% linen, (12 x 13 weft x warp)). Linen was chosen as it is typically / historically used as a lining fabric for the traditional studio adhesive formulations (described

below). Linen was also used for the 'Mist-Lining' Plextol adhesive mock-ups. The third canvas was a modern polyester canvas called Clipper (Maritex) (24 x 24 weft x warp) [5]. Polyester canvases have been used as a lining support since the 1980s, especially when combined with BEVA 371 O.F.[™] lining systems. The simulation of the original painting were primed artist canvases. All used material combinations are listed in table 1.

Adhesive	Lining canvas	Dummy painting	
Glue Paste	Rough canvas	Oil primed linen canvas (Peter van Ginkel). Type A	
Wax Resin	Rough canvas	Oil primed linen canvas (Peter van Ginkel). Type B	
BEVA 371	Clipper canvas	Oil primed linen canvas. Studio formulation.	
Plextol	Fine canvas	Oil primed linen canvas (Claessens)	

 Table 1. Overview of the combination of the researched canvases and adhesives.

2.2. Adhesive formulations for lining Four different adhesive formulations were used for the lining tests: **GP:** The procedure followed that practiced at The National Gallery London. This mixture is composed of 2g animal glue, 12g wheat flour and 72ml water.

WR: Traditional GP linings tend to be moisture sensitive and thus prone to attack by micro-organisms. The recipe used for this experiment is based on that provided by The National Gallery, London conservators. It consists of beeswax, dammar and gum elemi in the ratio: 3:2:1. Different recipes are prevalent in different geographical areas.

BEVA-copolymer: One of the most established synthetic multi-purpose adhesives in the art conservation is BEVA 371, which is a synthetic co-polymer of ethylene and vinyl acetate, developed by Gustav Berger. [6, 7, 8] The content of ethylene in the copolymers amounts to 60-80% which is thus the main co-monomer. The properties of EVA depend on the ratio of ethylene and vinyl acetate. Less than 30% vinyl acetate shows a partially crystalline thermoplastic formulation. In contrast, a content of 40-70% vinyl acetate causes a rubber-like and amorphous formulation. All in all, vinyl acetate reduces the crystallinity of EVA and increases the solubility making it more polar. The more vinyl acetate, the higher the flexibility, density and absorption capacity for additives. The other components are included to modify the melt temperature and application method.

Plextol mixture: acrylic based adhesives used in this study were prepared in a mixture of Plextol D541 and K360 with two drops of Rohagit SD15. The mixture consisted of a 30/70 ratio. The pH of the more acidic Plextol K360 was modified prior to mixing to avoid any adverse chemical reaction when combining with the more alkaline Plextol D541. Additional formulations with an increased proportion of the higher molecular weight Plextol adhesive D541 will be tested in the future.

Brush outs / films from each adhesive mixture were caste onto MelinexTM sheets and used for experiments.

3. Methods

3.1 Application methods for lining A different methodology was used to adhere the secondary canvas to the reverse of the dummy painting. Each technique used to create samples mirrored actual practice.

GP Lining: This lining technique is first recorded in the early-to-mid 17th century in France and Italy. It is still widely practiced although drawbacks are well disseminated. [9] The lining canvas is tightly loomed, washed and re-stretched. The dummy painting is left unrestrained. The front of the dummy painting is faced with a sheet of Japanese paper adhered, in this case, with a diluted solution of BEVA 371 O.F.TM. The glue-paste is applied thinly to the back of the dummy painting and the lining canvas by hand. Subsequently the lining canvas is placed on top of the dummy and the package is flipped to work face-up. An absorbent paper is placed beneath the lining canvas. The front is ironed at about 45°C, airing the structure at intervals, until the moisture has evaporated. The method followed is practiced at the National Gallery, London.

WR Lining: The wax resin lining was inspired by the preservation of mummies in the Egyptian tradition. [10] The wax resin lining system was introduced in the Netherlands in mid 19th century to combat these drawbacks, [11] and rapidly spread to become one of the most prevalent techniques to treat structural issues a century later. The front of the dummy painting is faced with a sheet of Japanese paper adhered with a diluted solution of wax-resin adhesive. The dummy painting is loomed using paper tensioning strips to a working loom. The lining canvas is loomed on to a stretcher larger than the working loom. The mixture is melted in an *au bain-marie* pan, and the warm wax resin is applied with a wide brush to both the reverse of the dummy painting and that of the linen canvas independently in small sections. Subsequently, each canvas was ironed at 60°C, again independently to achieve an even layer. The two canvases are then placed together, with the wax-resin applied surfaces facing each other. The painting surface is protected with sheets of paper and ironed until the wax-resin is seen to penetrate through to the front of the lining canvas. This can be noticed by the darkening of the dummy painting. The method employed is typical of wax-resin hand linings.

BEVA Lining: The polyester lining canvas is stretched to a working loom. The warm un-thinned BEVA adhesive is applied on the lining canvas, only where the painting will be placed, by a roller to ensure an even layer. The reverse of the un-stretched dummy painting is placed on the BEVA layer of the lining canvas. The lining process occurs on a heated vacuum table (Elkom GmbH) with a pressure of 55mbar and a temperature of a heated table 68°C. The two canvases are placed together and placed under pressure before heat is applied. The pressure is retained until the heat has dissipated. The method employed is typical of working practice as described in Young and Ackroyd.

Plextol Mist-Lining: The lining canvas is stretched to a working loom larger than the dummy painting. The size of the dummy painting is marked on the lining canvas. The lining canvas is lightly sanded to enhance the nap. The acrylic lining adhesive is sprayed on to the lining canvas. The quantity of the adhesive for this case study was $74g/m^2$. The adhesive droplets adhere only to the upstanding nap of the lining canvas and does not penetrate the lining canvas weave. The water content is allowed to evaporate. This procedure ensures an open network of adhesive material which can be regenerated when placed behind the dummy painting in an enclosed envelop. The activation solvent is introduced in a controlled manner using a pre-set value ($60ml//m^2$ by dampening a delivery cloth and placing it behind the lining canvas. In this case Xylene was used. The two canvases are pressed together by low-pressure (20mbar) in an envelope. This method was developed at SRAL by Jos van Och [12, 13]. The result is a lining system in which the minimal amount of adhesive is required to ensure a bond and no adhesive impregnation of either the original or lining canvases.

3.2. Differential Scanning Calorimetry (DSC) In order to obtain the glass transition temperature (T_g) and softening or melting temperature (T_m) of the used adhesives, the differential scanning calorimetry technique (DSC) was utilised. DSC measurements were done on a TA Instrument DSC 250 equipment. The test samples, removed from the brush-outs, each weighed about 8 mg and were conditioned at 48% relative humidity at 22°C in a controlled climate chamber for 72 hours before being tested. The samples were cooled and heated in cycles. Each adhesive was subjected to a different temperature range profile depending where the thermal transitions occurred.

GP: The cooling cycle started at room temperature and sample was cooled to -20° C and held at this temperature for 5 minutes. Subsequently, the sample was heated up from -20 to 150°C. The temperature was held at 150°C for 5 minutes and the sample was cooled down to -20° C, and then this temperature was held for 5 minutes. Finally, the sample was heated to room temperature. A ramp of 10°C/min was chosen for both heating and cooling cycles.

WR: The sample was initially cooled from room temperature to -60° C and held at this temperature during 5 minutes. Subsequently, the sample was heated from -60° C to 150° C and held at this temperature for 5 minutes and subsequently cooled down to -60° C again. Then, the sample was heated up from -60° C to room temperature.

BEVA and Plextol: Similar heating and cooling cycles as for the wax resin were performed on BEVA 371 and Plextol samples.

3.3. Lap-shear test Lap shear tests were done according to the standard method ASTM D-1002-10 [14] with size modifications on a Zwick 20kN tensile test machine using a 1kN load cell for more accuracy. The aim of this test is to determine the apparent shear strength of adhesives. Sample dimensions are shown in figure 2. The samples were cut from each of the lined mock-ups.



Figure 2. Lap shear test sample dimensions based on ASTM D-1002-10. [14] (not on scale)

Figure 3. T-Peel test sample dimensions based on ASTM D-1876-01. [15]

3.4. T-Peel test T-peel tests were done according to the standard method ASTM D-1876-01 [15] with small size modifications. Test results give information on the relative peel resistance of the adhesive bond-to-peel loading and shows the weakest part of the adhesive bond as a whole. Figure 3 shows the substrate dimensions. These tests were conducted on a Zwick 20kN tensile test machine using a 1kN load cell. The separation rate was set to 50mm/min and at least 127mm lap length was peeled off. Samples were cut from each of the lined mock-ups.



3.5. Creep test setup The test setup is a laboratory built instrument, based on the spring-loaded apparatus as described in the ASTM D-2294 standard. [16] In this setup four different samples can be measured independently at the same time. The displacement of the samples is measured by a triangulation displacement sensor Opto NCDT from Micro-Epsilon in a range of 20mm and an accuracy of $4\mu m$, supplying every minute a data point to a computer database. The test setup was placed in a Weiss WK111 340 climate chamber The test set up is shown in figure 4.

The dimensions of the adhesively bonded samples are shown in figure 5. The sample sizes are based on ASTM D-1002 and ASTM D-5656-04 [17] with an overlap of 5mm only for each of the three lap-shear samples measured in series, in order to obtain an optimal flattened stress–strain distribution. Samples were obtained from each of the lined mock-ups.

4. Experimental results

4.1. Differential scanning calorimetry (DSC) Glass transition is the gradual and reversible transition in amorphous materials (or in amorphous regions within semi-crystalline materials) where the polymer changes from a hard and relatively brittle "glassy" state into a rubbery state as the temperature is

increased. At this second order thermodynamic transition (from glassy to a rubbery state) the modulus of elasticity reduces, which might lead to a premature failure during loading. Most polymer mixtures are made of incompatible polymers in which each polymer keeps its own individuality. As a result different T_g 's might be detected when measuring with differential calorimetry (DSC).

The identification of thermal transitions is very important, since they remarkably affect the mechanical performance such as creep behaviour of different adhesive/canvas systems. The glass transition temperatures and melting temperature range (from starting to finishing temperatures) associated with the endothermic melting peak are tabulated in table 2. In case of **GP**, the temperature range of the endothermic peak is related to the denaturation rather than a melting event. The thermal transition values of the various mixtures are quite different, with the lowest melting point for the **WR** material which starts melting at 25° C.

Sample Name	Wax Resin	Glue Paste	BEVA 371	Plextol
Tg (°C)	-	48°C	-28°C	-29°C
Tm (°C) range	25-27°C	79-90°C	30-90°C	43-60°C

 Table 2. Glass transition and melting temperature range for the different adhesives.

4.2. Lap shear tests The mechanical behaviour of the different adhesives has been compared, when bonded, to a reference canvas (pre-ground medium rough (pure) canvas (Rijn Uni) purchased from Claessens Canvas, Belgium) and tested in tensile loading on a 10kN Zwick test machine equipped with a 1kN load cell based on ASTM D1002. The loading speed was 1.3mm/min. The overlap was 12.5mm. Testing took place at laboratory conditions at that time (20°C and 60% RH). The aim of these tests was to determine the apparent lap-shear strength of the adhesives as a set-point for the creep testing.

The results of the lap shear tests are presented in figure 6. The presented curves are averaged from 3 individual test results. It shows the typical stress-strain curves of the discussed adhesive systems in shear. Clearly visible is the difference in mechanical behaviour of the different adhesive systems at a lower stress, though they all exhibit very low moduli of elasticity. At higher loadings **GP** shows clearly relatively high failure strength compared to the other adhesives. The 30/70 acrylic adhesive **Plextol** shows both the lowest modulus, plasticity and by far the lowest strength before failure occurs. EVA copolymer **BEVA 371** shows medium strength in combination with a high strain to failure at museum conditions. All samples failed cohesively inside the adhesive layer.

4.3. Creep measurements at different temperatures Sustained loading is common in adhesive bonding. All polymers tend to creep, though some more than others. Especially with non-crosslinked thermoplastic materials, such as the ones researched here, creep might be so extensive that bond failure



Figure 6. The stress – strain curves of the four discussed adhesives compared at 20°C and 60% RH.



Figure 7. Three typical Creep graphs taken from the measurements of Plextol at different temperatures at 48% RH and the same loading conditions.

might occur prematurely. Additionally, a continuous stress level and/or the absorption of moisture or temperature elevations can increase the polymer degradation and thus creep phenomena. [18, 19] The samples tested were films cast of each adhesive. Additional experiments are required to establish the performance of the laminate structure after lining. Thus, the data presented here represents the performance of the adhesive rather than the laminate structure. The results, therefore, do not take into account the penetration potential of the adhesive into the canvas substrates as a function of the lining. The authors would expect that as a lining adhesive the **WR** samples will perform badly as this adhesive penetrates throughout the structure during lining.

The creep data presented here (table 3) of the different adhesives is derived from tests carried out at different temperatures, but at a constant relative humidity of 48%. They are obtained by measuring the deformation of a sample, as a function of time, up to 11200 minutes (due to practical time restrictions). The specimens are loaded by a predetermined stress level, which is 10% of the failure load of the lapshear joint. This set of tests has the purpose to determine the creep resistance of the different types of adhesive as a result of a limited loading under standard museum conditions. [20, 21]

A typical creep performance of the **Plextol** sample in the climate room at 3 different temperatures, while keeping a constant 48% RH, is shown in figure 7. This graph clearly shows the temperature sensitivity of this laminate system on the creep behaviour. The sample exposed to 45°C even failed during the creep test before the rupture point at around 10 minutes.

The overall test results (table 3) show that **GP** performs best at museum conditions (least strain), but also at higher temperatures, showing no failure. Additionally, it is also able to carry the highest loading. **Plextol** and **WR** perform worst as a lining adhesive, being highly temperature sensitive. The **WR** adhesive fails after the shortest time at the highest temperature, which is not so surprising since its melting point starts at 25°C.

		Condition		
Type of adhesive	Loading (N)	25°C/48%	35°C/48%	45°C/48%
Glue Paste	(4kg)	No failure	No failure	No failure
		10.70%	13.10%	17.20%
Wax Resin	(1.6kg)	Failure	Failure	Failure
		575omin	1.3min	o.o2min
BEVA 371	(2kg)	No failure	No failure	No failure
		22.00%	9124min	2.5min
Plextol	o.6kg)	Failure	Failure	Failure
		400min	175min	10min

Table 3. Overview of the tested adhesives at different temperatures, at the same RH, including the loading and their overall performance If failure occurred within 11200 minutes, the time to rupture is given, otherwise the strain after 11200 minutes is presented. Green means an excellent performance.

5. Conclusions

Four lining adhesives were tested in a range of temperatures (20-45°C) at a relative humidity of 48%. The animal glue-wheat paste (**GP**) mocks-ups performed best, while beeswax-damar resin (**WR**) and acrylic **Plextol** mock-ups are found to be rather weak and more temperature sensitive. DSC measurements confirm this behaviour, with the glass transition for animal glue-wheat paste being above 48°C, whilst the other adhesives all show lower thermal transitions. **BEVA 371 O.F.TM** samples show the highest ductility at room temperature, but starts melting above 30°C. Finally, it should be noted that, while of vital importance, the mechanical performance of the lining adhesive is not the only consideration when choosing an adhesive for lining. The Mist-Lining system is a lining process in which the lining adhesive forms a true nap bond without any impregnation into either the lining or original canvases. This factor, combined with its mechanical behaviour described here, improves the potential reversibility of this system. This paper presents research on the compared thermal and mechanical responsive under load of prevalent lining adhesives for the first time using a standardised testing

procedure. The results will allow conservators to make better informed decisions when choosing lining adhesives, as selection criteria can now include performance related data.

Acknowledgements

The authors would like to thank the following people: interns: Lena Wesselink, Russell van den Beld, Nicolas Rocton, Mathilde Monachon and Mathieu Parnot. We want to thank Johan Bijleveld, Marlies Nijemeisland, Frans Oostrum, and Gertjan Mulder for training and making instruments available. All other laboratory staff: Fred Bosch, Victor Horbowiec, and the people from the TU Delft DEMO workshop.

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