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Adhesives for Wax Artifacts: Investigation of Suitable Materials and Their Adhesion Properties via Tensile and Bending Tests

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Abstract

Research on suitable adhesives for wax artifacts was necessary to carry out conservation treatment of the moulages at the German Hygiene Museum in Dresden. Due to the complexity of the collection, waxes of various chemical compositions had to be considered. Therefore, test specimens of beeswax, Japan wax, paraffin wax, and montan wax were prepared and treated with nine selected adhesives, including synthetic resins, polyethylene glycol, and animal glue. Tensile and bending tests on the test samples clearly showed that the diverse wax types place different demands on adhesives and that numerous other factors (e.g. the nature of the fracture surface, the size and weight of the broken pieces, and the possibility of holding the broken components stable while the adhesive cures) are critical in the choice of adhesive. The investigations identified three adhesive materials that are suitable for wax artifacts, but could not distinguish one that would be suitable in all cases. Rather, the final choice will always depend on the conditions specific to the object.

Titre et Résumé

Adhésifs pour objets en cire : étude de matériaux adéquats et de leurs propriétés d'adhérence au moyen d'essais de traction et de flexion

Il était essentiel d'exécuter des travaux de recherche sur les adhésifs adéquats pour le traitement d'objets en cire, afin de pouvoir par la suite réaliser la restauration de moulages de la collection du German Hygiene Museum de Dresde. La nature très complexe de la collection exigeait de tenir compte de la présence de cires ayant diverses compositions chimiques. Des échantillons d'essai de cire d'abeille, de cire du Japon, de cire de paraffine et de cire de lignite ont donc été préparés et traités avec neuf adhésifs particuliers, dont des résines synthétiques, du polyéthylèneglycol et une colle animale. Les résultats d'essais de traction et de flexion effectués sur les échantillons indiquent clairement que les divers types de cires exigent l'emploi d'adhésifs possédant différentes propriétés et que de nombreux autres facteurs (par exemple, la nature de la surface de rupture, la taille et le poids des morceaux de l'objet, ainsi que la possibilité de joindre et de stabiliser les composants pendant que l'adhésif durcit) constituent des éléments cruciaux lors du choix du meilleur adhésif. Les résultats des essais ont permis d'identifier trois matériaux adhésifs pouvant servir à restaurer adéquatement des objets en cire, mais ils ne permettent pas de déterminer si un de ceux-ci peut être utilisé dans tous les cas possibles. Le choix final reposera donc toujours sur l'état de l'objet à restaurer et les conditions de conservation ou d'exposition qui lui sont propres.

Introduction

This study is based on research and conservation treatments carried out on the moulage collection of the German Hygiene Museum in Dresden (Deutsches Hygiene-Museum Dresden, henceforth DHMD) within the framework of the project "Wax Moulages: A Valuable Handicraft Threatened with Extinction." Approximately 2000 moulages are preserved in Dresden, a great number of which require stabilisation with adhesives because of fractures in the wax (e. g., Figure 1).

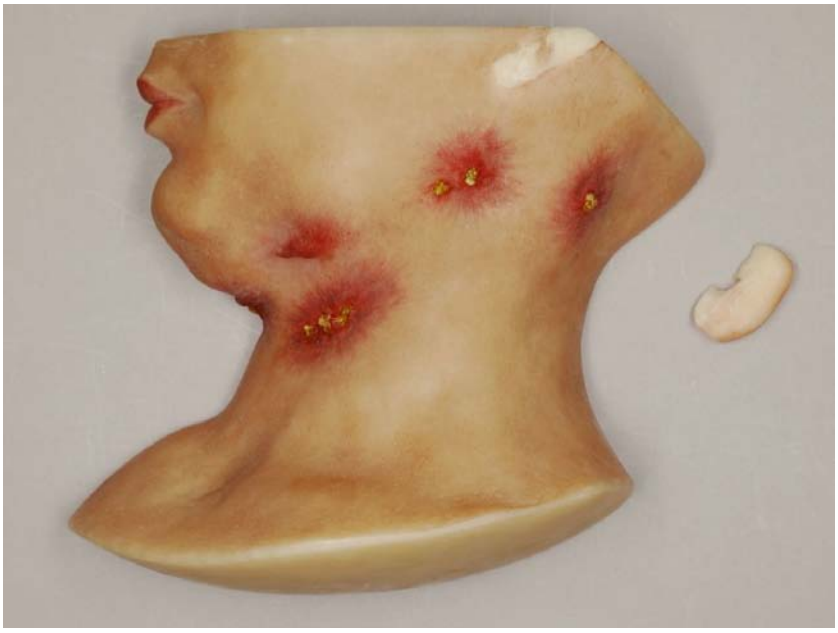

Figure 1. Wax moulage (DHMD 1995/607) with fracture, before conservation treatment.

Wax presents unfavourable conditions for adhesion. Poor wettability, low polarity and a smooth surface prevent the formation of strong intermolecular and mechanical bonds at the interface between the fractured component and the adhesive (Habenicht 2006, pp. 297-299, 332-336, 647-649). The sensitivity of wax to heat and solvents severely limits the choice of materials suitable for achieving effective bonding, since many of the adhesives used in conservation are applied warm or as solutions. Effective adhesion is also difficult to achieve because it is generally impossible to hold broken wax pieces securely while the adhesive cures.

The following study investigates suitable adhesives for wax artefacts and is based on previous works by Murell 1971; Kaufmann 1998; Fillip 1998; Hierl 2000; Grausam 2002; Raddatz, and Fischer 2003; Reifarth 2003; Kokarnig 2004; Stremmel 2006; Wittstadt 2006; Eska 2009. Whilst these studies primarily consider mainly beeswax as the common material for wax sculptures, the present paper will for the first time include other types of wax, too.

The Wax Moulages at the German Hygiene Museum Dresden

Moulages are three-dimensional wax reproductions of pathologically affected human body parts. They were originally used as teaching aids in medical training and as visual material for laymen in health education (Schnalke 1995). The DHMD was an important center for the production of moulages since its foundation in 1912 until the 1980s. Mixtures of waxes, plant resins and chalk were the base materials processed in Dresden, and besides beeswax also carnauba wax, Japan wax, montan wax and paraffin wax were used (Walther-Hecker 2010, pp. 154-155).

Experiments to Identify Suitable Adhesives

On specially made wax samples selected adhesives were tested for their suitability. The chosen test methods were tensile and bending tests, since they would produce forces similar to those that might affect historic wax artefacts. Adhesives that showed suitable properties in the tests were then used for the conservation treatment of the Dresden moulages.

Selection of adhesives

In the above mentioned papers, primarily aqueous dispersions of synthetic resin, polyethylene glycol and animal glues are recommended as adhesives for wax, and rarely synthetic resins dissolved in organic solvents. For this study, representatives of the first three groups of adhesives were chosen initially, as they comply best with general conservation requirements (sufficient adhesive strength with a certain amount of elasticity, so that no tension would endanger the join or the intact material; chemical and physical stability with ageing, e.g. permanent adhesive strength, no embrittlement, no yellowing, no releases of harmful substances; permanent reversibility; curing as transparent film; well-known ingredients etc.) and the particular demands of the material wax (above all application without the use of heat or solvents, since this would harm the wax; fast curing, since wax artefacts do not allow clamping during the adhesive cures; removability of excess adhesive and long-term reversibility of the cured adhesive with water and without great mechanical force, since this would endanger the wax). The aim was to achieve a tensile and bend strength with the adhered samples that would not be higher than the strength of the reference samples which were not treated with adhesive since this would indicate that the wax artefact would break in the material substrate before the adhesive joint would fail. In the meantime, the adhered samples were meant to perform only a slightly lower strength as the reference samples since otherwise the adhesion would not guarantee to hold the treated wax artefact fast enough together.

Following preliminary experiments regarding the application properties and drying behaviour of various products, a total of four adhesives were subjected to tensile tests (Figure 2). These tests showed that for some wax-types none of the adhesives were suitable since they did not reveal sufficient strength. Hence, the selection of adhesives had to be altered to include stronger synthetic resins although these had to be applied dissolved in solvents. The identification of appropriate synthetic resin solutions in turn required solubility tests on wax test samples as well

as trials using various adhesive preparations. Five adhesives were finally subjected to bending tests (see Table 1).

Table 1. Adhesives selected for tensile and bending tests.

Adhesive	Trade Name	Chemical Composition	Application Specification
Tensile tests			
1	Mowilith DMC 2	poly(vinyl acetate); aqueous dispersion of a copolymer based on vinyl acetate and maleic acid di-n-butyl ester	applied as ready-made product without further dilution
2	Polyethylene glycol 6000	high molecular weight polymer of ethylene oxide	applied as a 60 % (W/W) solution in deionised water + ethanol (1+1)
3	Lascaux 498 HV + Lascaux 360 HV	poly(alkyl acrylate); aqueous dispersions of a thermoplastic acrylic polymer on the basis of methyl methacrylate and butyl acrylate; thickened with acrylic butylester	applied as ready-made products mixed in a 1+1 ratio (W/W)
4	Lascaux 498 HV		applied as ready-made product without further dilution
5	Isinglass + wheat starch	collagen (isinglass); carbohydrate (wheat starch)	applied as a 1+1 mixture (W/W); with the isinglass as a 30 % solution, and the wheat starch as 20 % solution in deionised water
Bending tests			
1	Paraloid B 72	ethyl methacrylate co-polymer	applied as a 40 % solution (W/W) in acetone + ethanol (1+1)
2	Mowilith 30	poly(vinyl acetate); polymer of vinyl acetate	applied as a 40 % solution (W/W) in ethanol + deionised water (1+19)
3	Mowilith 50		
4	Isinglass	collagen	applied as a 30 % solution in deionised water (W/W)
5	Lascaux 498 HV	poly(alkyl acrylate); aqueous dispersion of a thermoplastic acrylic polymer on the basis of methyl methacrylate and butyl acrylate; thickened with acrylic butylester	applied as ready-made product without further dilution

Production of test samples

In order to represent the diverse composition of the DHMD moulages, six different mixtures of components were prepared for production of the test samples (Table 2). Recipes from the Dresden moulage workshop provided guidance for the selection of the raw materials and their ratio, as did the results of a material analysis carried out on selected moulages (Dietemann et al. 2010, pp. 65-73). Consideration was also given to the results gained from melting temperature measurements, which indicated that the DHMD collection includes both soft and hard moulages.

Table 2. Composition of the test samples.

Wax mixture	**Raw materials**	**Ratio**
1	Japan wax Dammar resin Chalk (calcium carbonate)	80 % 10 % 10 %
2	Beeswax, bleached Colophony (pine resin) Chalk	80 % 10 % 10 %
3	Paraffin wax; soft Paraffin wax; hard Carnauba wax Colophony Chalk	32 % 40 % 20 % 4 % 4 %
4	Paraffin wax; soft Paraffin wax; hard Carnauba wax Colophony Chalk	44 % 35 % 13 % 4 % 4 %
5	Esterwax on montan basis; “soft” Paraffin wax; hard Colophony Chalk	73 % 17 % 5 % 5 %
6	Esterwax on montan basis; “soft” Montanic acid wax; hard Paraffin wax; hard Colophony Chalk	49 % 29 % 14 % 4 % 4 %

According to the historic process of moulage-making, the waxes were melted separately in a water bath and mixed together whilst still liquid (Walther-Hecker 2010, p. 156). Then, the resin was added, which had been melted under high heat, followed by the addition of finely ground chalk. The resulting mixtures of low viscosity were poured into elastic silicone moulds, from which the hardened test samples could be removed without mechanical stress. To avoid faulty casts with unequal thickness and air bubbles, two-part moulds were used and the wax mixtures were poured through a funnel (Figure 2).

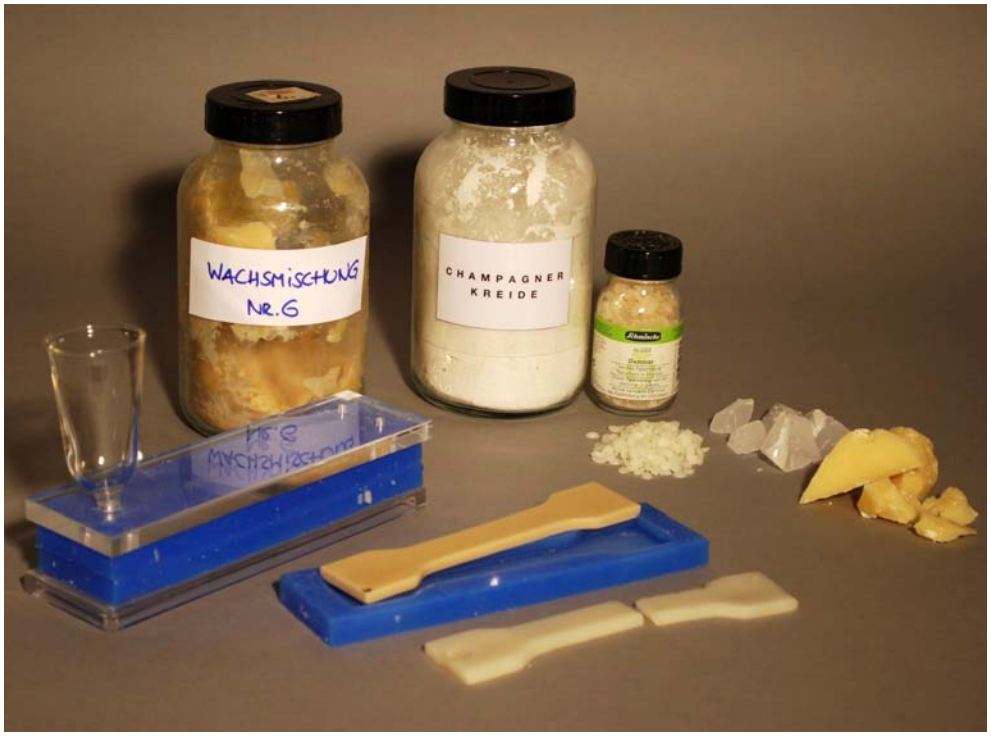

Figure 2. Material used for making the test samples.

Although there are standards for the tensile and bending testing of materials that include specifications of test sample shape and size (e.g., DIN EN ISO 178, E DIN EN ISO 527-2), no standards exist so far for the testing of wax. Therefore, suitable test specimens for wax had to be developed based on the available standards and on initial experiments. For the tensile tests, dumb-bell shaped samples with dimensions of 170 mm x 20 mm x 6 mm proved to be suitable (Figure 3), and for the bending tests, bar-shaped samples of 60 mm length and 10 mm diameter (see Figure 4).

Figure 3. Dumb-bell shaped samples for tensile tests.

For each wax mixture, a set of reference samples was tested to gain the average strength value for the material. These samples were neither fractured nor treated with adhesive. Further sets were fractured, then treated with the selected adhesives and tested under identical conditions. Although the testing of a large number of samples generally improves the precision of the mean strength values measured, only three reference samples of each wax mixture as well as three samples for each adhesive could be produced for the tensile tests due to a limited availability of material. After analysis of the tests, the samples were melted again and reused for production of the bending test samples. The smaller dimensions of the latter enabled the manufacture of seven samples per wax mixture and adhesive, and seven reference samples (Figure 4).

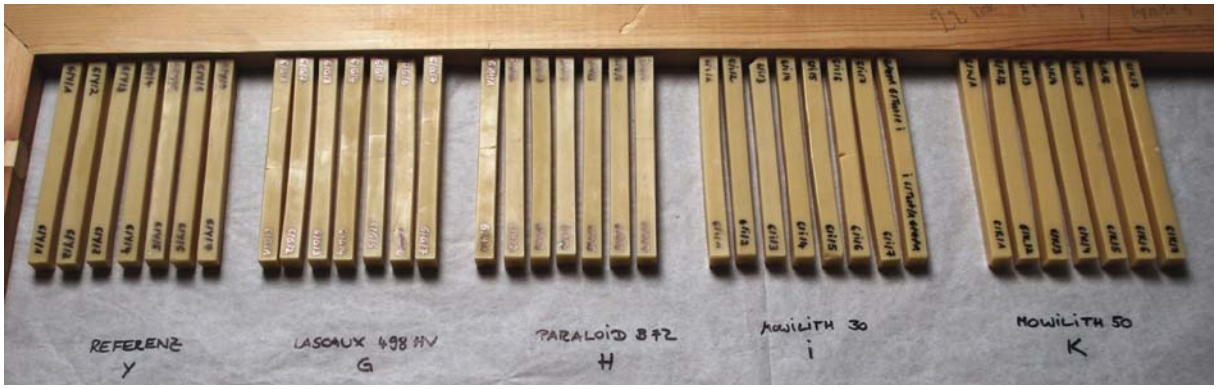

Figure 4. Bar-shaped samples for bending tests.

Conditioning and fracture of the test samples

The test samples were stored for three weeks at c. 20 °C and 50 % RH. Measurements of the melting temperature carried out after conditioning showed that the samples displayed similar values to those of the moulages. A longer storage period would have been desirable given that wax embrittles with time, but this was not possible due to time constraints. However, as the brittleness of wax is a significant parameter influencing its fracture behaviour, it had to be produced "artificially." For this purpose, the samples were placed in a freezer for ten minutes, and immediately afterwards broken by hand. The fractures thus produced appeared very similar with those on the moulages.

Adhesion of the test samples

The adhesives were applied with a brush to both fracture surfaces of the samples; the isinglass was applied lukewarm. A gentle, manually applied compression of the broken pieces for three minutes followed hereafter, before the samples were stored again for two weeks under the aforementioned conditions to guarantee complete curing of the adhesive.

Experimental details

The tensile tests were carried out on a Zwick material testing machine equipped with a 2.5 kN load cell (Figure 5). The testing rate was 200 mm/min. The vices grips of the testing apparatus were covered with sandpaper in order to prevent slippage of the samples during testing.

Figure 5. Wax sample during tensile tests.

For the bending tests, the machine was equipped with a three-point bending tool (Figure 6). The span width of the support was 60 mm. The load cell and testing rate remained the same as for the tensile tests.

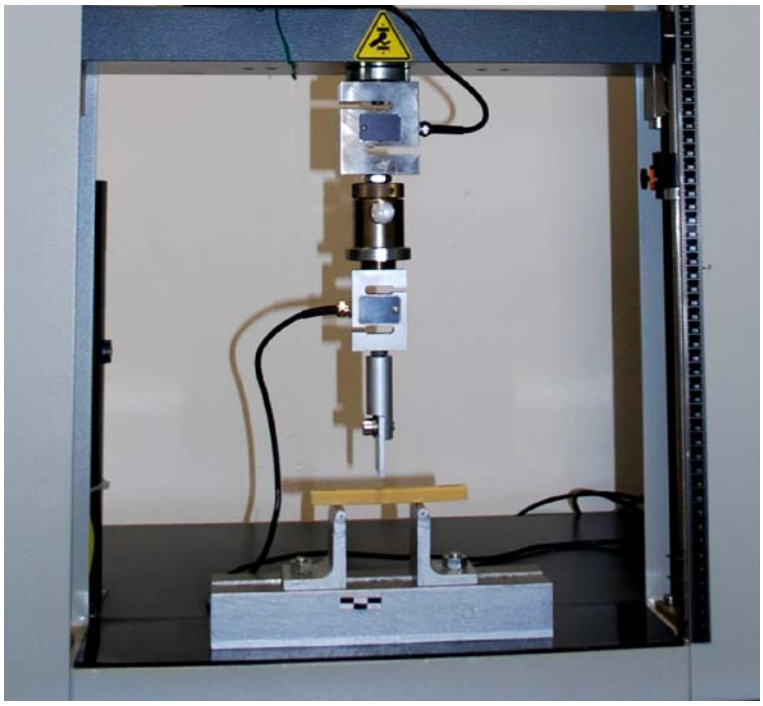

Figure 6. Wax sample during bending tests.

Results and Discussion

Despite some deviations, clear trends could be identified from the values measured for the tested specimens and enabled an assessment of the various adhesives. The essential results of both test series are presented below.

Tensile tests

The tensile tests showed that the diverse wax-types required different adhesives due in particular to a different nature of fracture surface and therefore a modification of the selection of adhesives was necessary for goal-oriented implementation of the bending tests. A schematic comparison of the strength value of the reference sample with those measured after adhesive application makes this particularly clear (Figure 7).

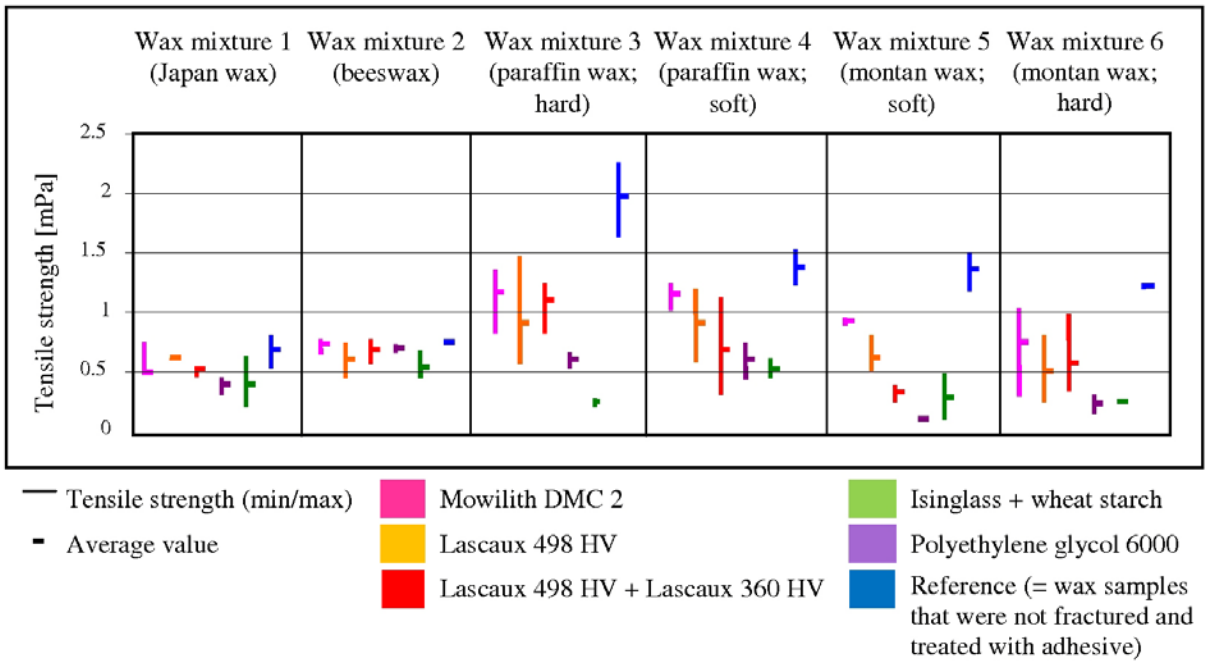

Figure 7. Results of tensile testing.

On both the Japan wax and beeswax samples adhered with Mowilith DMC 2, the adhesive strength values came very close to and in a few cases even exceeded the strength of the reference samples. On the paraffin wax samples, the adhesive strength of the Mowilith DMC 2 also proved to be slightly too high since in some cases breakage occurred in the wax and not within the adhesive joint. However, the main disadvantages of the poly(vinyl acetate) dispersion are its tendency to yellow with increasing age (Howells et al. 1984, pp. 29-30, 33; Down et al. 2009, p. 95) and the fact that according to the manufacturer it is no longer produced (Galla 2010). Given these problems, Mowilith DMC 2 cannot be considered as suitable adhesive for wax and therefore was not included in the bending tests.

Polyethylene glycol 6000 (PEG 6000) proved to be too weak to adhere any of the waxes, because the strength of the re-adhered samples was always far below that of the strength of the reference samples. The low adhesive strength was particularly clear on the samples made of paraffin and montan wax, which often readily broke during clamping in the testing machine. Moreover, PEG 6000 application proved problematical when dissolved in pure water because of its very poor wetting of the wax surface. Although this problem could be counteracted by dissolution in a mixture of ethanol and water, this was of problematic approach as the resin contained in the wax mixtures is soluble in alcohol. Therefore, PEG 6000 was also not included in the bending tests.

By mixing the non-tacky curing Lascaux 498 HV with the permanently viscoplastic Lascaux 360 HV, the properties of both acrylic dispersions were combined, in order to optimise their adhesive performance. However, no advantages over pure Lascaux 498 HV could be detected, and since the production of Lascaux 360 HV is to be discontinued (Fritschi 2009), the mixture of the two products was not included in the bending tests.

On the beeswax samples which were treated with pure Lascaux 498 HV, a necking sometimes appeared close to the adhered joint, indicating too high adhesive strength. On the montan wax samples the adhesive strength was too low, i.e. nearly all specimens showed strength values below the strength of the reference samples. In contrast, the Japan and paraffin wax samples always broke at the adhesive joint and their strength reached similar values to that of the reference samples. Thus, indicating the suitability of Lascaux 498 HV for these cases, it was tested further with bending experiments.

A mixture of isinglass with wheat starch paste was expected to yield better wetting of the wax surface, which would encourage the formation of effective bonding. However, for the paraffin and montan wax samples the adhesive strength of the mixture turned out to be very low nonetheless: the samples mostly broke during clamping. In contrast, the mixture showed effective bonding on the beeswax and Japan wax samples, whose strength was closer to the strength of the reference samples and which always broke at the adhesive joint. Only a slight increase in strength proved desirable here, and hence for the bending tests the component with the greater adhesive strength, i.e. the isinglass, was used by itself.

Bending tests

For the bending tests, adhesives were individually selected for each wax mixture according to the conclusions drawn from the tensile tests. To some extent this led to ideal results, as explained in the following. A diagram with the measurements shows that the strength of the samples after adhesion was always significantly below the strength of the reference samples (Figure 8). As this was the case even when fracture occurred within the adhesive joint, the results raised new questions in this respect. Nevertheless, it was possible to discern tendencies concerning the suitability of the tested materials for adhering wax artefacts.

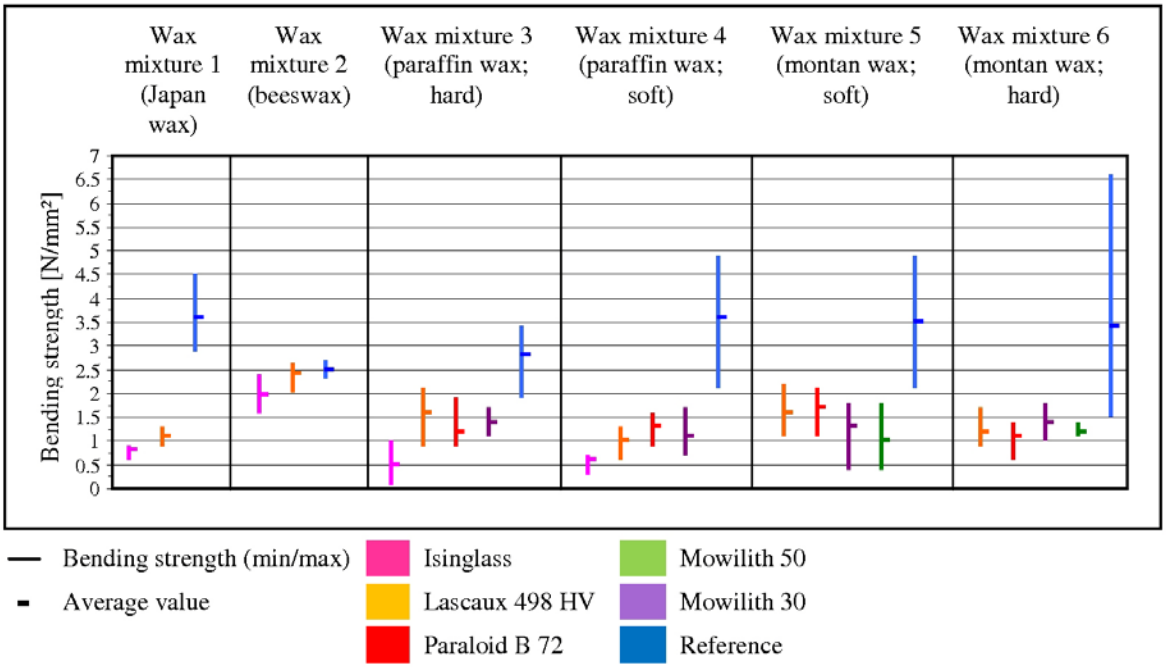

Figure 8. Results of Bending tests.

Adhesion with Paraloid B 72 dissolved in acetone induced fractures equally in the wax and within the adhesive joint and the test samples exhibited greatly deviating strength values. Only a conditional assessment of the adhesive was possible based on these varied results. Unfavourable application and drying behaviour, in particular fast evaporation of the solvent which impedes uniform wetting of the fracture surfaces, limit its use. Moreover, since the solvent was suspected of causing certain changes to the fracture surface (Figure 9), the Paraloid B 72 solution in acetone appeared unsuitable in the final assessment.

Figure 9. Fracture surface after bending test; arrow indicates glossy area where the wax surface has softened probably due to the influence of the solvent of the adhesive.

The Mowilith 30 likewise produced greatly varying results, so that it could not be judged on the test data alone. However, the measurements showed a trend towards higher strength values, as required particularly by montan waxes. Accordingly, the poly(vinyl acetate) was considered for this type of wax, since it is a solution in ethanol-water which is less likely to cause damage on wax than a synthetic resins dissolved in a stronger solvent. Moreover, for wax artefacts with sensitive surfaces the relatively rapid drying of the Mowilith 30 proved advantageous; thus it was not necessary to hold together the fractured sample parts for a long time. However, despite these positive properties, the use of Mowilith 30 is merely a compromise because it is only reversible mechanically or with solvents and hence can endanger the wax.

Likewise, the more viscous Mowilith 50 produced varying strength values, although in general these were lower than those from Mowilith 30. The reason for this was the irregular behaviour of the adhesive in the course of the drying process, during which the adhesive joints mostly reopened. To prevent this adhesive failure, it would be necessary to support the fracture joint for a prolonged time until the adhesive is fully cured. Unfortunately, however, this practice is not possible on soft wax mixtures, some of which proved to be very sensitive to wrapping with tape or plastic foil as support. Therefore, Mowilith 50 cannot be considered a suitable adhesive for those waxes.

Isinglass proved unsuitable as adhesive for paraffin wax because the strength of the glued samples was clearly below that of the reference samples. A comparable deviation was observed

for Japan wax, although this must be considered with reservations regarding the positive results which the mixture of isinglass and wheat starch paste achieved during tensile testing. For beeswax, the values were only very slightly below the strength of the reference samples and the fracture always occurred within the adhesive joint. Thus isinglass is to be recommended as adhesive for beeswax. Good application and drying properties further support its use. The rapid initial drying of the isinglass means that the broken pieces have to be held together for only a short time. Its lasting solubility in water guarantees permanent reversibility, without affecting the wax. Moreover, excess isinglass can be removed at a later stage, so that the adhesive can dry thoroughly without exposure to mechanical stress.

The samples adhered with Lascaux 498 HV frequently broke in the wax and not in the adhesive joint, indicating its unsuitability. Moreover, its slow drying-speed, which requires prolonged support of the pieces during initial curing, must be considered, again particularly regarding the softer wax mixtures, which are sensitive to the slightest contact. Additionally this adhesive is problematic for all waxes due to its insolubility in water once cured. Excess adhesive therefore has to be removed shortly after application. This exposes the joint to stresses before the adhesive has cured. Despite these disadvantages, use of Lascaux 498 HV for certain wax artefacts should be considered nonetheless. Broken components that are themselves quite heavy require a greater adhesive strength than can be achieved with isinglass. Because of its longer open-time compared with the rapidly drying isinglass, the Lascaux 498 HV can be applied better to large fractured surfaces. Hence, its use as an adhesive may be a suitable compromise.

Conclusion

The tests to determine suitable materials for adhesion of wax artefacts revealed that different wax-types require different adhesives. According to the results of the tensile tests, montan and paraffin wax mixtures require high adhesive strength, whereas for beeswax and Japan wax adhesives with lower strength suffice.

The test results and the practical knowledge gained on the behaviour of wax artefacts during the investigations further showed that several factors are critical in the choice of an adhesive. Not only is the wax type important, but also the nature of the fracture surface, the size and weight of the broken pieces as well as the possibility of holding the broken components stable while the adhesive cures.

In light of these findings it becomes clear that no single adhesive can be recommended across the board for conservation treatment of wax artefacts. Rather, three materials proved to be suitable in the course of these investigations. However, the choice must also depend on considerations of conditions specific to each particular object.

For works of art that consist primarily of beeswax preference should generally be given to isinglass. This choice is based equally on the positive results of the bending tests and the advantageous application and drying properties of the glue. Furthermore, isinglass can always be removed with water, and long-term experience in conservation shows good aging

characteristics of this adhesive. The adhesion of heavy fractured pieces or that of smooth fracture surfaces made of beeswax requires a greater adhesive strength than isinglass can provide. The same applies to the adhesion of paraffin and montan wax artworks. Greater adhesive strength can be achieved with Lascaux 498 HV, although this adhesive only dries slowly and therefore stabilisation of the readhered components is required until fully cured. This renders the adhesive unsuitable for soft waxes and those with very sensitive surfaces, which may be easily damaged whilst supported. In such cases, Mowilith 30 should be preferred, which hardens much faster. However, like Lascaux 498 HV, it is insoluble in water when cured, and therefore is only reversible mechanically or through the use of stronger solvents. Since this constitutes great danger for wax artefacts, the use of synthetic resin represents merely a compromise.

Wax objects can differ considerably in their fracture behaviour, as shown in particular by the varying results of the bending tests. Too many factors determine their properties, including the chemical composition of the wax, the way it was processed, and environmental conditions during aging. Therefore, adhesive selection has to be decided individually for every object. This test series enabled the identification of helpful reference points for making these choices, and their first application within the conservation treatment of the moulages from the DHMD led to positive results (Figure 10).

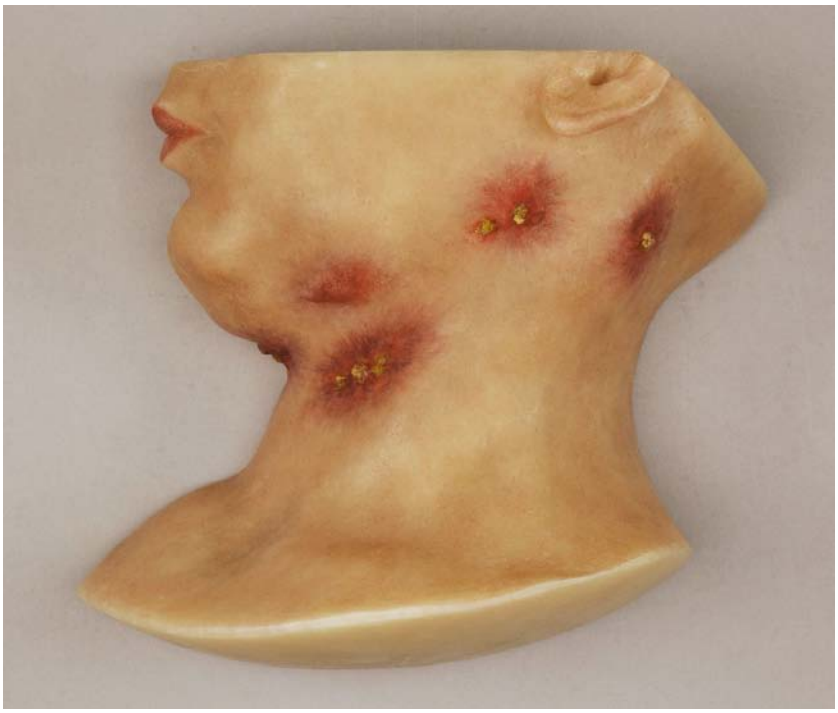

Figure 10. Wax moulage, after conservation treatment.

This study clearly indicates the unlikelihood that future research may lead to the identification of a uniformly suitable adhesive for all wax artefacts. Nonetheless, further investigations are desirable in order to ascertain adhesives that are even more suitable than the ones already tested. Such investigations should include further tensile tests using those adhesives that showed good results in the bending tests. Furthermore, the execution of both types of experiments on aged

wax test samples is desirable. Together with other studies in this field, the identification of the strength properties of adhesive joins on wax promises to be a useful starting point for further research in the field of wax conservation. In addition, the search should be continued for identifying whether pure wax exhibits a different behaviour to adhesives than the tested mixtures made of wax, resin and chalk, and whether the recommendations for adhesive selection must be adjusted accordingly.

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Materials and Suppliers

Beeswax, bleached. Fa. Kremer Pigmente; http://www.kremer-pigmente.de.

Carnauba wax, bleached. Fa. Kremer Pigmente; http://www.kremer-pigmente.de.

Chalk from Champagne. Fa. Kremer Pigmente; http://www.kremer-pigmente.de.

Colophony extra light. Fa. Kremer Pigmente; http://www.kremer-pigmente.de.

Dammar resin, best quality. Fa. Kremer Pigmente; http://www.kremer-pigmente.de.

Esterwax on montan basis, "soft": TeCe-Wachs RD 703 T (72-78 °C). Wachs- und Ceresin-Fabriken Th. C. Tromm GmbH; http://www.wax-tromm.de.

Isinglass. Salianski. Fa. Kremer Pigmente; http://www.kremer-pigmente.de.

Japan wax: Fa. Gerhard Eggebrecht, Süderau/Germany.

Lascaux 360 HV; Lascaux 498 HV. Acrylic Glue. Deffner & Johann GmbH; http://www.deffner-johann.de.

Montanic acid wax, hard: Montanwachs 30850 S (78-82 °C). Wachs- und Ceresin-Fabriken Th. C. Tromm GmbH; http://www.wax-tromm.de.

Mowilith 30; Mowilith 50; Mowilith DMC 2. Poly(vinyl acetate). Fa. Kremer Pigmente; http://www.kremer-pigmente.de.

Paraffin wax, soft: Polarit Z 40 (38-42 °C); Paraffin wax, hard: Polarit 30789 (60-62 °C). Wachs- und Ceresin-Fabriken Th. C. Tromm GmbH; http://www.wax-tromm.de.

Paraloid B 72. Ethyl methacrylate polymer. Fa. Kremer Pigmente; http://www.kremer-pigmente.de.

Polyethylene glycol 6000. Polyoxyethylen. Carl Roth GmbH + Co.KG; http://www.carlroth.com.

Silicone casting compound Duplosil DM, fluid, Liquid A und B. Deffner & Johann GmbH; http://www.deffner-johann.de.

Wheat starch. Local Pharmacy, Dresden.

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