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PROTOTYPES OF LASCAUX'S MEDIUM FOR CONSOLIDATION

DEVELOPMENT OF A NEW CUSTOM-MADE POLYMER DISPERSION FOR USE IN CONSERVATION

In 2004 Lascaux Colours & Restauro, supplier of conservation materials based in Switzerland, marketed for the first time a new polymer dispersion product known as "Medium for Consolidation". This material was made to consolidate polychrome sculptures and other types of painted objects.

"Medium for Consolidation" is seen as an effective replacement for the BASF product Acronal 300D which was used for this purpose until manufacture ceased in the mid-1990s. This paper describes the origin and development of Lascaux's "Medium for Consolidation" and the stability test performed on two prototype products.

BACKGROUND

During the 1960s, a wide range of proprietary synthetic polymer materials were introduced into conservation for use as adhesives, consolidants and coatings. One of them was Acronal 300D, an acrylic polymer dispersion produced by BASF, Ludwigshafen. The industrial application of this material was to laminate paper, but in conservation Acronal 300D was found to have special properties which rendered it particularly suited to consolidate flaking paint layers on polychrome wooden sculpture. Although a wide range of acrylic dispersions with similar chemical properties was available on the market at the time, Acronal 300D proved to have unique working properties, of which

its impressive capillary penetration was perhaps the most significant and distinctive. Acronal made it possible with a minimum of risk, to consolidate painted and water-sensitive gilt surfaces on chalk ground as well as distemper on wood without pre-treating the surface. Acronal penetrated without difficulty through minute cracks. Due to the adhesive's short drying time, softening of otherwise extremely water-sensitive paint layers could be avoided. Acronal could be used on animal-glue-based paint and water gilding despite its high water content and the fact that it was often used further diluted. If any excess consolidant was left on the surface it could be removed with acetone or xylene.

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Flemish altarpiece from Villberga Church, Uppland, Sweden. Around 40 early 16th century Flemish altarpieces with their polychromy largely intact can still be found in Sweden.



Photo: G. Hildebrand

In the 1960s, Acronal 300D was tested at the Landesmuseum Zurich, with good results.¹ However, Acronal 300D came to be used far more in Sweden than elsewhere in Europe due to its special conservation needs. In Sweden, medieval polychrome wooden sculptures and altars with original polychromy intact have been preserved to a greater extent than in any other country outside Germany. A majority of these objects can still be found in churches throughout Sweden. After World War II, most Swedish churches installed central heating which led to generally much lower indoor humidities. The effect inevitably was that all painted wooden objects that had been kept in a fairly stable and high humidity for centuries soon showed massive flaking of the paint layers. A conservation survey in the late 1970s revealed that about 90% of all painted wooden objects in Swedish churches were in need of immediate treatment.

ANIMAL GLUE AND WAX

The situation was further complicated by the fact that previous treatments using excessive amounts of animal glue had created severe tension in the paint layers. Previous treatments with wax, also, had impregnated matt surfaces, thereby drastically changing their appearance. Accordingly, the continued use of animal glue or wax for consolidation was regarded as unjustifiable. Rather, it was felt that what was required was a mechanically benign consolidant that effectively secured even humidity-responsive layers of polychromy without changing the appearance of the object. In addition to severe flaking, most objects also were covered with dust and dirt. With this in mind it was of utmost importance to use a material with better properties than the traditional ones, such as animal glue and wax, that had caused such devastating problems in the past. As in most cases, the surface dirt on these objects were impossible to remove before treatment, it was necessary to use a consolidant that could be removed from extremely water sensitive surfaces without any further damage.

SYNTHETIC CONSOLIDANTS

Using a synthetic consolidant on polychrome sculpture was not a new idea. Dispersions such as PVA had been widely used in post war Germany. Quite a number of Germany's most important sculptures had been treated with PVA. However, it became apparent that Acronal 300D worked so much better than PVA mostly because of its physical properties in a liquid state. Acronal 300D had a very small particle size, extremely low viscosity and low surface tension which conferred very good capillary flow properties which enabled it to penetrate almost any paint layer. During the course of 25 years several hundreds of medieval polychrome sculptures and altarpieces as well as later painted



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Detail of the Villberga altarpiece

wooden objects in churches were treated with Acronal 300D. The problems connected with the consolidation of such a vast amount of severely damaged objects could not have been solved with any other consolidant or with any other method.

FURTHER EVALUATION OF ACRONAL 300D

In 1994 it was decided to perform further evaluations of Acronal 300D. The manufacturer BASF, was contacted in order to obtain new information about the content of the material. Concerns had been expressed by some conservators about the presence of vinyl chloride as one of the monomers and about the possible long-term implications for the stability of the polymer itself and any object treated with it. The Royal Institute of Technology in Stockholm carried out new ageing tests.² These tests and examination of objects that had been previously treated with Acronal revealed no evidence of any significant causes for alarm.^{3, 4} Slight tendency to yellow and the formation of acidic degradation products and insoluble material on ageing were noted by Phenix et al. in their stability tests of Acronal 300D and other low viscosity dispersions.⁵

However, about the same time production of Acronal 300D ceased, as it was no longer any industrial demand. Attempts to persuade BASF to continue small batch production of Acronal for conservation purposes only proved futile, as it was not financially justifiable for the company to produce such small quantities. Efforts were made to find a similar product already on the market.⁶ A wide range of products with similar properties were tested by us, but none of these were found to have the working properties of Acronal. In the absence of any suitable existing commercial polymer dispersions, contact was made with Lascaux Colours & Restauro,

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Photo: Peter Tångeberg

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Baroque altar, circa 1700.
Detail showing damages caused
by consolidation with animal glue

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Object treated with wax in the
early 1950's

a well-known firm in Switzerland that produces artist materials and conservation materials. After a meeting with the chemical research staff at Lascaux in which we discussed our needs and the reasons why we could not use any other acrylic resin already on the market, they kindly agreed to try to prepare a dispersion medium that fitted our needs and specification.

CRITERIA SETUP FOR THE MEDIUM FOR CONSOLIDATION

The following criteria were set up for the replacement product for Acronal 300D:

- The adhesive must be able to penetrate into the finest craquelure or microscopic openings in the paint and /or ground layers: it will be of low viscosity.
- It must have sufficiently low surface tension to enable it to wet the paint layer.
- It must form a macroscopic layer with sufficient elastic strength to withstand possible future dimensional variations of the (wooden) substrate.
- It must not form any degradation products that might be harmful to the object.
- It must not cause undesirable changes to the surface appearance of the paint or polychromy.
- It must be stable to the effects of light, temperature and humidity.
- It must be able to be applied and handled using the usual methods for consolidation of polychrome sculptures and other painted objects, such as brush or syringe.
- It must be possible to remove any excess adhesive with solvents that are relatively harmless to the object.



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- It must not cause any environmental hazards or be a threat to the health or safety of the user.
- It must have a pH within the normal range that would be considered acceptable for use on organic materials.

Following our discussions with Lascaux, the company provided a range of samples for practical evaluation. After two years of testing we finally decided upon two samples that seemed to have the required working properties. These two samples, hereafter called Exp. 5 and Exp. 6, were selected to be tested for stability by The Royal Institute of Technology, Stockholm. The test methods used corresponded to those used previously in 1995 for the stability testing of Acronal 300D.

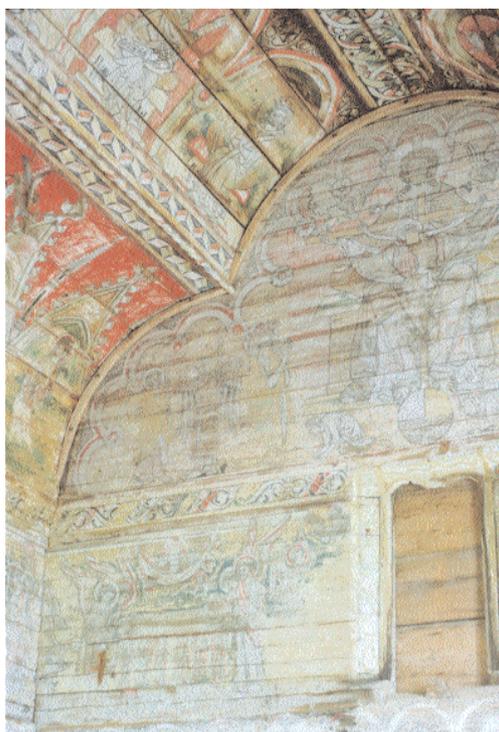
MATERIALS: COMPOSITION OF EXP. 5 AND EXP. 6 COMPARED TO ACRONAL 300 D

Table 1 presents a summary of the polymer compositions of Exp. 5 and Exp. 6 as supplied by Lascaux. Data on Acronal 300 D from other sources is added for comparison. Further chemical analysis of Exp. 5 and Exp. 6 by Pyrolysis Gas Chromatography/Mass Spectrometry (Py-GCMS) confirmed these two products as styrenated acrylics: both samples had similar monomer constituents, which were identified as butyl acrylate, methyl acrylate, methylmethacrylate and styrene.⁷ Since they lead to the formation of relatively soft, low Tg polymers, the acrylate monomers can be regarded as internal plasticizers. By contrast, both methylmethacrylate and styrene lead to high Tg polymers and effectively produce an internal stiffening effect on the polymer backbone.

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As can be seen, the monomer composition of Acronal 300D and of the two new alternatives differs significantly. The new systems are based on monomers with arguably better properties compared to Acronal 300D: the vinyl chloride present in Acronal 300D does not occur in Exp. 5 and Exp. 6. Acrylate and methacrylate monomers are generally considered to be very stable and resistant to degradation. Sentence deleted here: you were pre-determining the outcome of your experiments. Perhaps the only real question concerning Exp. 5 and Exp. 6 is related to the possible presence of styrene monomer. Styrene monomer may degrade under UV-irradiation and with loss of mechanical properties and tenden-



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cies to yellow. The effect of this can be directly related the amount of styrene present and have to be evaluated by ageing experiments/stability tests.

ADDITIVES

Like most polymer dispersions, Exp. 5 and Exp. 6 also contain small amounts of various additives (3%) and solvents (2%). The additives are non-ionic surfactants, defoamers, and a fungicide. The presence of solvent (typically glycol ethers) in Exp. 5 and Exp. 6 is necessary in order to promote good film formation: the dry films should be homogeneous and the dispersed polymer particles should have coalesced properly. The solvent itself will have

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Typical flaking caused by low relative humidity due to central heating. Additionally this Madonna is covered with dust.

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Distemper paintings from 1323 in the wooden church of Södra Råda. These paintings were treated with Acronal 300D between the years 1981–83.

Property	Acronal 300D	Exp. 5	Exp. 6
Polymer composition**	Acrylic-terpolymer	Acrylic copolymer	Acrylic copolymer
Elasticity	Hard	Hard-elastic	Elastic
Penetration in:			
Filter paper	Very good	Very good	Very good
Old distemper	Very good	Excellent	Excellent
Old wood surface	Good	Good	Good
Minimum Film Formation Temperature (MFT)	Approx. 17°C	Approx. 4°C	Approx. 4°C
Colour stability	Yellowing	Clear transparent film	Clear transparent film
Weather stability	No	Yes	Yes
pH-value	4,5–6	8,4 ± 0,2	8,4 ± 0,2
Average particle size	~0,3 µm	~0,03–0,3 µm	~0,03 – 0,3 µm
Dilutability with water	Any desired	Any desired	Any desired
Soluble in:	Ester solvents Aromatic solvents Acetone, MEK	Ester solvents Aromatic solvents Acetone, MEK	Ester solvents Aromatic solvents Acetone, MEK

** More details on the composition of the polymers are presented in table 2.

Tab. 1

Table 1
Comparison of Properties of Acronal 300D, Exp. 5*, and Exp. 6* (* Information supplied by Lascaux.)

Tab. 2

Monomer type	Acronal 300D	Exp. 5	Exp. 6
Acrylate ester	+	+	+
Vinyl acetate	+		
Vinyl chloride	+		
Styrene		+	+
Methacrylate ester		+	+

Tab. 3

Exposure time (hours)	Colour		Opacity		Surface erosion	
	Exp. 5	Exp. 6	Exp. 5	Exp. 6	Exp. 5	Exp. 6
0	None	None	Clear	Clear	None	None
500	None	None	Slightly	Slightly	Very little	Very little
1000	None	None	Slightly	Slightly	Little	Little
1500	None	None	Slightly	Slightly	Yes	Yes

Table 2
Monomer types present in Acronal 300D, Exp. 5, and Exp. 6

Table 3
Visual evaluation data on aged samples of Exp. 5 and Exp. 6

little effect on long-term durability since it evaporates during drying. The effect of the solvent is instead seen on the film forming properties where the MFT is as low as 4°C. According to the producer, the glass transition temperatures of the two new polymer dispersions are in the region of 4°C.⁸

The additives that exist in Exp. 5 and Exp. 6 cannot be omitted for technical reasons: they are essential ingredients for the formation, stability and film-forming properties of the polymer dispersions. The additives should not significantly affect the mechanical performance of the final film and thus not change the properties if they evaporate, migrate or react chemically with time. The only question is whether the additives will degrade and form harmful or discoloured compounds with time. An ageing study will reveal any tendency to discolour. The additives are used in such small amounts that they may be considered as very low risk components in the system.

STABILITY TESTING AND EVALUATION

EXPERIMENTAL AGEING PROCEDURES

Dispersions Exp. 5 and Exp. 6 were applied as thin coatings on aluminium plates with a 60 mm film applicator and let to dry overnight in room temperature. The films were then subjected to accelerated ageing in a QUV Accelerated Weathering Tester

from Q-Panel. The ageing sequence consisted of: 4 minutes of water spray, 4 hours at 50°C above liquid water (humid atmosphere), and UV-A-irradiation at 60°C for 4 hours with an intensity of 0.63 W/m². This sequence was then repeated for periods of 500, 1000, and 1500 hours respectively.

EVALUATION PROCEDURES

VISUAL EVALUATION

The aged films were evaluated with respect to yellowing, deterioration, cloudiness or other physical changes by visual examination. The flexibility of the aged samples was also judged qualitatively when removing small specimens from the panels.

IR-SPECTROSCOPY

Small specimens of the films were removed from the test panels and studied with FTIR-spectroscopy to determine if any chemical changes had occurred. FTIR measurements were performed on a Perkin Elmer Spectrum 2000 FTIR spectrophotometer equipped with a single reflection ATR-accessory from Specac Ltd

MOLECULAR WEIGHT DETERMINATIONS

Small samples were removed from the panels, dissolved in dimethyl formamide (DMF) and characterized with respect to molecular weight by Size Exclusion Chromatography (SEC). Molecular weight determinations were performed on a Waters SEC instrument using dimethylformamide (DMF) as a solvent and linear polyethyleneglycol standards for calibration.⁹

RESULTS

GENERAL COMMENTS

The accelerated ageing conditions used here represent, in the order of things, a very severe test of stability and probably correspond to very long exposures under normal outdoor conditions or to indoor conditions where there is no real environ-

Figure 1
Schematic diagram of samples used in accelerated ageing tests. This diagram could be made much clearer – some shading to indicate the sample film would help, for example.

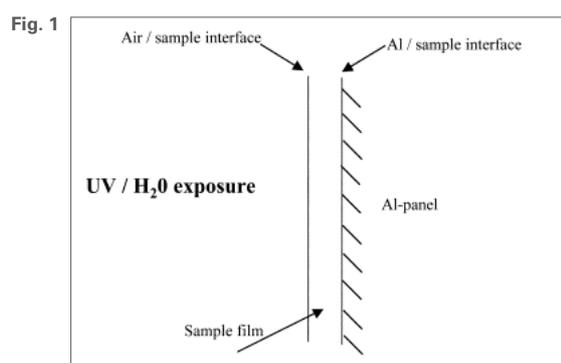


Fig. 1

mental control, as might occur in churches. The combination of light irradiation, water spray, humid atmosphere, and elevated temperature together represent a very harsh environment that will demonstrate the resistance of the test material(s) to photochemical, hydrolytic and, to some extent, thermal deterioration. While it is appreciated that these severe exposure conditions would not occur in the intended use for Exp. 5 and Exp. 6 as a consolidant for painted or polychromed objects, it is believed that, if they withstand the test, it can be safely assumed that they will be stable in the foreseeable future in the intended application.

VISUAL EVALUATION OF THE AGED SAMPLES

Table 3 presents the results of the visual evaluation of the different aged samples. The degradation observed appears to proceed mostly as surface erosion and is most probably due to the combination of water exposure, high humidity and elevated temperature. No significant colour changes were observed in the polymer films, which indicates that Exp. 5 and Exp. 6 are quite resistant to yellowing caused by exposure to high dosage of light. The colour stability of these materials is even better than that of Acronal 300D. The polymer films tend to become slightly opaque on ageing, but this is mainly due to the surface erosion making the surface slightly more rough/matt. A rough surface scatters light making the appearance of the film slightly opaque. The aged films of Exp. 5 and Exp. 6 were observed to be clear and transparent beneath a very thin surface layer. It is assumed that the opacity/roughening of the exposed surface of the polymer films that is caused during exposure in the QUV Weathering tester is more due to hydrolytic than to photochemical degradation processes. The surface erosion is expected to be associated with the loss of low molecular weight degradation products and an increase in surface polarity.

Although assessed purely subjectively, no changes in flexibility could be detected when removing the films from the panels. Both before and after ageing, Exp. 6 was more flexible than Exp. 5: this property might make Exp. 6 preferable as a consolidant to Exp. 5.

IR-SPECTROSCOPY

Small pieces were removed from the films using a scalpel. The measurements were then made in reflection mode (ATR-technique) on the pieces. The aged films have two surfaces, of course, one directed towards the substrate (i.e. the aluminium panel) and one facing the atmosphere and light source during ageing, as shown in Figure 1. With the ATR-technique a surface layer of just a few microns of the film is actually analyzed: the IR-

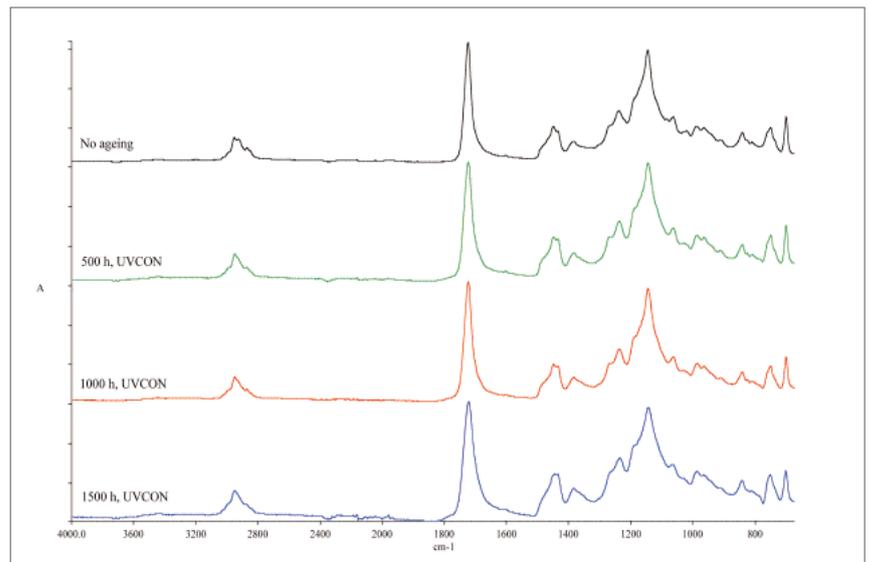


Fig. 2

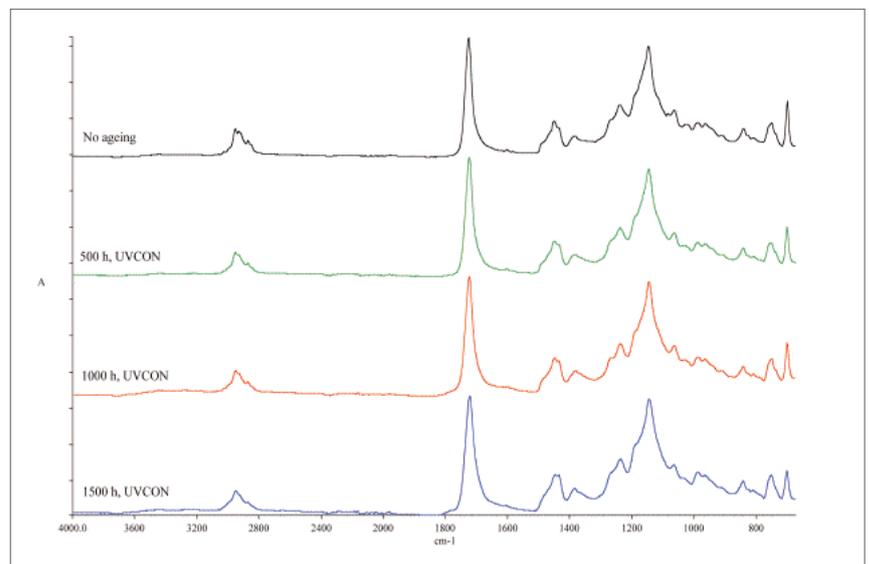


Fig. 3

spectrum corresponds only to a small fraction of the total film thickness. Comparing spectra from the two surfaces would show if there are any differences in degradation through the thickness of the film. Accordingly, both sides of the aged films were analyzed using this technique.

Figure 2 presents IR spectra on all samples of Exp. 5 determined on the surface facing the aluminium panel during ageing. Virtually no difference can be discerned between the spectra indicating that no or very little degradation had occurred at that surface. Chemical alteration/degradation would be indicated by differences between the spectra obtained after different times of exposure in the QUV Weathering tester. The results are similar for Exp. 6 (see Figure 3) indicating very similar behaviour of Exp. 5 and Exp. 6. Both are polymers that are resistant to degradation under the test conditions.

Figure 2
IR-spectra of Exp. 5 samples, surfaces facing the aluminium panel. Samples aged for 0, 500, 1000, 1500 hours respectively in the QUV Weathering tester

Figure 3
IR-spectra of Exp. 6 samples, surfaces facing the aluminium panel. Samples aged for 0, 500, 1000, 1500 hours respectively in the QUV Weathering tester

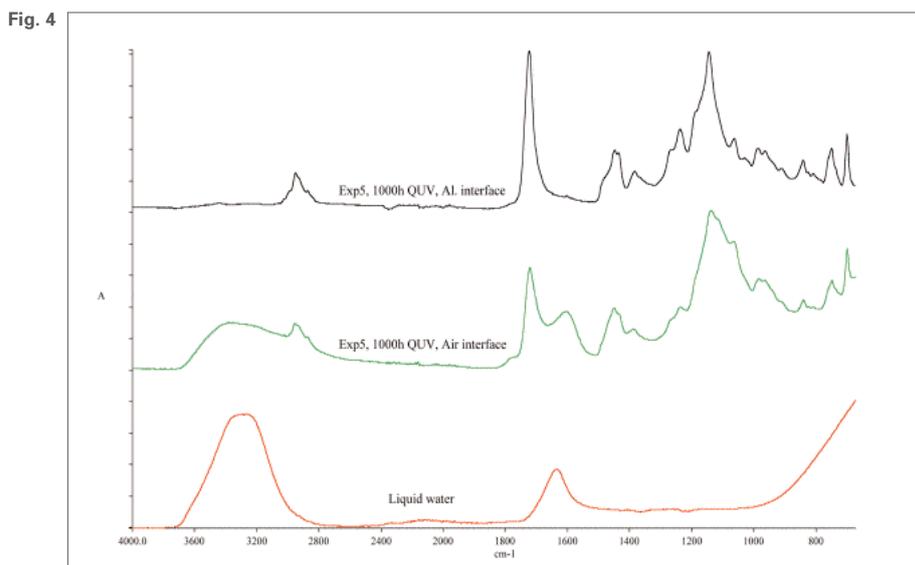


Figure 4
IR spectra of Exp. 5 exposed for 100 h in the QUV-tester. Spectra obtained at the air and the aluminium interface respectively. Spectrum of liquid water added at the bottom

Figure 4 presents two spectra of Exp. 5 aged for 1000 h. The difference between the spectra is that the spectra are taken from the two different sides of the aged film. There is a great difference between the spectra where the one placed towards the atmosphere has large peaks at around 3200 and 1600 cm^{-1} . These peaks resemble the absorption spectrum of water (also in Figure 4) indicating that water is strongly absorbed at the interface. The differences are in accordance with the reasoning that the surface erosion is due to a certain amount of hydrolysis at the surface in a humid atmosphere at elevated temperature and exposure to UV-light. This degradation mechanism is highly unlikely to occur in the intended use of Exp. 5 and Exp. 6.

MOLECULAR WEIGHT DETERMINATIONS

The investigation of possible changes in molecular weight during the ageing process encountered some technical difficulties either respect to the intrinsic solubility of the polymers, which meant that detailed results could not be obtained. All samples proved rather difficult to dissolve even in strongly polar solvents such as DMF. However, some qualitative observations can be made with reasonable confidence.

Unaged samples of Exp. 5 and Exp. 6 and samples of the polymers aged for 500 and 1000 h respectively were dissolved in DMF, the solutions filtered and then characterised with SEC. It could be concluded from the SEC analysis that the polymers are of very high molecular weight (>300 000) and that high molecular weight polymers also are present after the ageing process. This supports the previous discussion that degradation proceeds via a surface erosion process under the very severe ageing conditions employed.

DISCUSSION

The present work is intended to be an evaluation of Exp. 5 and Exp. 6 based on the demands and knowledge described above as a replacement to Acronal 300D.

An accelerated ageing study on both resins has been performed to evaluate the long-term durability. A general discussion is also made concerning the differences between Acronal 300D, Exp. 5 and Exp. 6. A water based polymer dispersion was considered to be the prime target as replacement for Acronal 300D since the concept has a successful history. Two replacement alternatives, Exp. 5 and Exp. 6, were tested.

A real disadvantage of most synthetic consolidants, and a much more relevant one than the fact that they have unpredictable ageing properties (which is not entirely true), is however the fact that they were generally not made for conservation purposes at all, but for some industrial or household purpose. Therefore, we can never be sure that in the products are not altered nor is there any guarantee of future supply, because the products can quite simply disappear from the market.

It proved difficult to choose between the tested products but in the end Exp. 6 was selected for its elasticity. However, similar to Acronal D 300 Exp. 6 also proved useful for consolidating weakly bound distemper on wood as well as on fabric, diluted with distilled water, with or without wetting with white spirit. As Acronal D 300 proved to be useful for other purposes than the originally intended this probably also for the case with Exp. 6.

It may well be that no new consolidant is the final solution for our problem consolidating paintlayers on all kinds of materials, but to our knowledge there is no other binding medium on the market at the moment that to a higher extent meets the highest ethical and technical demands that can be put on a binding media used on priceless objects of art than Experiment 6. Experiment 6 can now be found in Lascaux's catalogue under the name of "Lascaux Medium für Konsolidierung" (MFK).

Lascaux is producing this binding medium is being produced by Lascaux directly corresponding to our high demands for a binding medium used on polychrome wooden sculptures and knowing that the conservation world is a small market.

CONCLUSIONS

The article describes the background of the use of Acronal D300 and its important role in the recent history of Swedish conservation. Acronal D300 was a water dispersion of polymer latex particles. The polymer in Acronal 300D was a copolymer based on vinylchloride, vinylacetate and n-butyl acrylate.

The setback due to the termination of production of Acronal D300 had ceased made it necessary

to begin research to find a substitute product. It was decided to find a product with the working properties of Acronal but without the vinyl chloride in the manufacturing process. Lascaux Colours & Restauro produced a range of samples that were practically evaluated by conservation professionals. The most suitable alternatives were subjected to ageing tests as reported in the article.

Two different water dispersions of acrylate polymers were aged and studied with respect to accelerated degradation. Both polymers are based on acrylate monomers and, contrary to Acronal 300D, do not contain any vinyl chloride monomers. The dispersions contain only minimal amounts of additives to avoid any problem caused by the migration of low molecular weight fractions. The dispersions do not contain anything that could be hazardous when handling them. Films of the polymers have been subjected to accelerated ageing in QUV-equipment. The results show that both polymers exhibit good stability to degradation. IR-analysis of films after ageing shows that the polymers only degrade to some extent due to surface erosion and do not degrade throughout the film. This indicates that they will be very stable in the intended application since the conditions in this case are rather mild. Surface erosion such as the one seen in the accelerated ageing will only occur under very harsh conditions. The study further shows that both polymers are non-yellowing and retain their appearance with time.

The mechanical properties differ slightly between the dispersions: Exp. 6 is more ductile than Exp. 5 at ambient conditions. Due to the different ratios of the monomers in the acrylate polymers and not due to any degradation.

Both polymers exhibit very good stability and can be considered as very good candidates for the intended purpose. Preference between the two

dispersions is mainly determined by the desired flexibility and not stability issues.

The results also propose that the degradation proceed via a mechanism that cannot occur in the intended application for the consolidation material. SEC-analysis of the aged materials gives only qualitative results but they support the aforementioned IR-study. All tests indicated that this was a highly stable acrylic resin with extremely good ageing properties.

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- ⁶ Ironically, the Röhm GmbH product, Plextol D528, that performed very well in the stability tests of Phenix et al, and which was proposed by that group as an alternative to Acronal 300D, was also discontinued from supply in the late 1990s.
- ⁷ GCMS analysis kindly carried out by Bronwyn Ormsby at Tate, London 2004. Py-GC-MS was performed on a GSG Pyromat Curie Point pyrolyzer interfaced to a Varian Saturn 2000 GC-MS instrument. Pyrolysis conditions: 625°C for 10 seconds. GC conditions: held at 40°C for 2 minutes then ramped to 320°C at 10 C/minute, then held for further 2 minutes at 320°C. MS conditions: EI mode (70 eV); scanned 40-399 amu every 0.49 seconds.
- ⁸ The glass-transition temperature, the T_g, is the temperature above which a polymer becomes soft and rubbery. This transition is normally given a specific value for simplicity reasons but it is important to note that it is a simplification. The T_g-transition is not a narrow transition but occurs over a temperature range with a gradual decrease in stiffness of the material. Both the width and position of the transition varies from polymer to polymer. The T_g is also rate dependent i.e. a material may appear stiff at one temperature when slowly deformed. This altogether means that great care must be taken when evaluating the effect of the T_g on mechanical properties of a polymer.
- ⁹ Tetrahydrofuran (THF) and chloroform (CHCl₃) were also tested as solvents for the SEC analysis.

APPENDIX: SUPPLIERS' DETAILS

Analytical instruments:

The ageing was performed with a QUV Accelerated Weathering Tester from Q-Panel. FTIR measurements were performed on a Perkin Elmer Spectrum 2000 FTIR equipped with a single reflection ATR-accessory from Specac Ltd. Molecular weight determinations were performed on a Waters SEC equipment using DMF as solvent and linear polyethyleneglycol standards for calibration.

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