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THE INFLUENCE OF ORGANIC SOLVENTS ON THE MECHANICAL PROPERTIES OF ALKYD AND OIL PAINT

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ABSTRACT

Organic solvents are often used to assist in the removal of varnishes and overpaints, however their effect on the properties of paint films has not yet been well established. The effects of organic solvents on the mechanical properties of alkyd and oil paint are here examined. Alkyd paints were very popular in the 1950s, when artists like Jackson Pollock stated: "New needs need new techniques". The solvent response of household paints based on oil-modified polyester resin is compared with that of artists' alkyd paints and oil paints. Swell tests on paint strips suggest that all paint systems respond to the same solvents, but alkyd paints are less sensitive. Leached components were characterised by FTIR. As expected, more polar solvents tend to extract more polar paint components. To detect the changes in the mechanical properties, stress-strain measurements were performed before and after solvent treatment of the different pigmented paint strips. These clearly show that alkyd and oil paints become more stiff and brittle due to treatment with different solvents.

Although the extraction of polar and softening components has an influence on the mechanical properties of paint films, as widely reported, the results shown here demonstrate that it does not seem to be the only factor responsible for the mechanical changes. The swelling power of solvents and the extraction of low molecular components are just two factors. Other properties like evaporation rate have an important influence on the solvent-induced degradation of alkyd and oil paint films. Moreover, our results suggest a more complex degradation mechanism of the paint film when exposed to solvents, not simply explained by the extraction or swelling phenomena. The results reported here will help the conservator to estimate the potential harm of organic solvent-based conservation treatments like the removal of varnishes or overpaints.

USE OF ALKYD PAINTS IN ART

Alkyd resins are oil-modified polyester resins developed in the late 1920s [Küchenmeister 1978]. The first commercial alkyd resin-based paint was produced under the name "Glyptal" in 1926 in the United States by General Electric [Derrik et al. 2005]. They have been widely used in the European surface coatings industry since the 1950s. Since then, alkyd resins have replaced the drying oils in many industrial applications because of their shorter drying time and higher resistance properties [Schilling et al. 2004]. At the end of the 1950s, the first development phase was concluded and alkyd resins had, in terms of volume, become the most important raw material for synthetic coating resins. As a result of their popularity as household paints, these products naturally also became accessible to artists. Pablo Picasso was one of the first artists to experiment with synthetic paints. Cappitelli et al. [2005] has shown that Picasso was using such paints back in 1932 (Fig. 1).

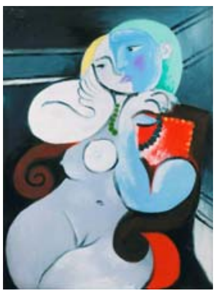

Fig. 1: Pablo Picasso: "Nude Woman in a Red Armchair", 1932. Alkyd resin-based paint on canvas.
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"New needs need new techniques", said Jackson Pollock in 1950 [Crook et al. 2000]. The characteristic effect of his famous "drip paintings" is based on the use of household and industrial paints. Pollock used a variety of paint materials for his "Summertime Number 9A" (Fig. 2) painted in 1948 [Learner 2006]. The fact that he selected his paints very carefully is shown by the detailed planning behind what, at first sight, is an extremely spontaneous looking piece of art.

Fig. 2: Jackson Pollock: "Summertime No. 9A", 1948. Oil paint and alkyd paint on canvas. The black and silver-grey paint that Pollock poured, dripped and sprayed unmixed with the brush handle onto the lying canvas is an Alkyd resin-based paint. These areas are noted for their smooth and glossy finish that is typical of such paints. Between the black and silver-grey areas, Pollock used shining coloured dabs of artists' oil paints. These carefully positioned areas differ significantly from the others in their visible brush flow, surface appearance and physical properties. © 2008, ProLitteris, Zürich / Tate, London

In the 1950s and 1960s, alkyd paints opened up new painting options for artists such as Peter Blake, Cy Twombly and Frank Stella. These paints fitted in perfectly with their search for new statements and techniques [Crook et al. 2004]. Apart from their optical appearance and short drying time, the social significance of alkyd resin as an industrial product and its low price also contributed to the increasing popularity of these paints in the art world.

Even though alkyd paints have declined in popularity since the 1970s, they are still used. The best-known contemporary representative is Damien Hirst. In contrast to household paints, artists' paints based on alkyd resin did not become particularly widespread. The painting and technical properties of the alkyd-based artists' paints manufactured by Winsor & Newton since the 1980s basically conform to those of oil paints, and are used more for traditional layer painting.

COMPOSITION AND DRYING OF ALKYD PAINTS

Alkyd resins are produced through the condensation of polybasic carboxylic acids and polyhydric alcohols. The incorporation of monobasic fatty acids (origin: drying oils) produces a polymer with a lower degree of crosslinking more suitable as paint binder. These are called oil-modified alkyds [Kittel 1998]. The word alkyd is made up of the modules *alco*hol and ac*id* [Crook et al. 2000]. During their production, not all the hydroxyl groups are generally substituted with fatty acids. Because of this structure, alkyd resins can be modified both with polycondensates and radically with vinyl polymers. This results in an enormous variety of products with very different properties. In the paint sector and for artists' paints, unmodified alkyds based on phthalic acid polyesters with a relatively high content of oxidisable fatty acids have become established. Like traditional oil paints, alkyd paints dry by oxidation through a radical crosslinking process. It would therefore be logical to compare the properties of alkyd paints with those of oil paints.

EXPERIMENTS

The swelling of the paint layer treated with solvent gives rise to the leaching-out of low-molecular weight components. The leached-out components have softening characteristics. If they are missing, irreversible embrittlement of the paint layer will occur [Feller et al. 1985, Hedley et al. 1990, McGlinchey 1991, Tumosa et al. 1999, Erhardt et al. 2005].

A systematic series of tests was performed to check this theory. The aim was to determine the extent of the embrittlement of the paints, as may occur, for example, through the removal of varnish. The effects of organic solvents on various paint systems were therefore tested under identical conditions.

Materials

Alkyd paints for artists and household alkyd paints were compared with artists' oil paints. The paints selected were the "Normal Professional" oil paint from the firm H. Schmincke, the "Griffin Alkyd" alkyd-based artists' paint from the firm Winsor & Newton, and an alkyd-based paint for indoor and outdoor use from the DIY store, Migros-Genossenschafts-Bund. To assess the influence of the pigmentation on the sensitivity to solvents and on the mechanical properties, the paints were formulated in black, white, red, blue and yellow. Therefore frequently used pigments of various pigment classes were selected: The polycyclic phthalocyanine pigment, phthalocyanine blue (PB 15); the polycyclic aminoanthraquinone pigment, alizarin red (PR 177); the monoazo pigment, Hansa yellow (PY 3); titanium white (PW 6) and ivory black (PBk 9) (information taken from Manufacturers' labels). The exact pigmentation of the household alkyd paints was not further investigated. Paint films were produced five months before the beginning of the tests. The paints were applied by doctor blade to siliconised Hostaphan. Both the Griffin alkyds and the oil paints were diluted with Shellsol T to improve the brushing properties. The wet layer thickness of the films was 300 µm. The paint films were pulled from the film for the tests and cut to size.

Methods

Swell test

The swelling of paint films immersed in solvent is regarded as an indirect measure of the solvent sensitivity. The swelling behaviour was therefore examined with 50 solvents covering the entire polarity spectrum. The solvents were selected in line with trials carried out by Alan Phenix [2002] and Stefan Zumbühl [2005]. This paper deals only with the results from seven solvents that are characteristic of the different solvent categories. The polarity of the selected solvents increases from n-hexane (corresponds to white spirit) to toluene, diethylether, chloroform, acetone, ethanol and finally water.

Analysis of solvent extractable components

Apart from the swelling behaviour of the paint strips, the study also looked at what paint components were leached out of the white pigmented paint strips when immersed in solvent. The extracted substances were examined by Fourier Transform Infrared Spectroscopy (FTIR). To obtain the solvent extracts for FTIR analysis, the unsupported paint strips were immersed in solvent in a glass dish. The strips were taken out of the solvent after 6 minutes. After evaporation of the solvent, visible residues remained in the dish. They were weighed and analysed by a Perkin Elmer System 2000 µ-Infrared Spectrometer. For comparison, also the untreated paint strips were characterised by FTIR.

Stress-strain measurements

The mechanical properties before and after solvent treatment of the three given paint systems were recorded using a Zwick testing machine. For the tensile tests, the paint strips were cut into pieces 7.5 cm x 1 cm and removed from the siliconised film. The strips were inserted in each of the seven selected solvents for one minute. This immersion time is similar to that used in practice for restoration work. The strips were dried for four weeks in a standard room climate. A shrinking of the paint films was observed. The relative shrinkage was documented.

Every tensile test was repeated eight times. The clamped length of the strips was 2.3 cm. A Zwick Material-Prüfmaschine Z2.5 / TN1S was used and the software testXpert®.

Test method	Parameter	Information content	Examined paints	
Swell test	Swelling	Maximum solvent absorption and swelling rate of the paint films	- Griffin alkyd - DIY alkyd - Oil paint	Black, white, blue, red, yellow
FTIR	Absorption IR radiation	Composition of the paints and their solvent extracts	- Griffin alkyd - DIY alkyd	White
Zwick testing machine	Elasticity modulus	Elongation at break and tensile strength of the untreated and treated paint strips	- Griffin alkyd - DIY alkyd - Oil paint	Black, white, blue, red, yellow

Tab. 1: Overview of the tests.

RESULTS

Swell Tests: Swelling Effect of Seven Different Solvents

In the swell test, the expansion of an unsupported paint strip in a solvent bath is measured [McCrone 1991, Phenix 2002, Zumbühl 2005]. The aim of the swell test is to ascertain the quantitative sensitivity to solvents of the various paint systems and colours. When a paint strip is immersed in solvent, it absorbs a limited amount of the surrounding liquid, and its volume increases accordingly. The heavier the swelling of the paint strip, the higher its sensitivity to solvents. The extent of the swelling process is governed essentially by the intermolecular interactions between the paint strip components and the solvent. These values give an indication of the potential sensitivity of the material in preservation work.
The pigmentation determines the binder requirement and has a major influence on the drying process and the mechanical properties of fresh paints. Consequently, it is to be expected that the pigments also exert an influence on the sensitivity to solvents. Paint strips pigmented with titanium white proved to be insensitive because the media contains components that accelerate the drying process. Films containing organic pigments are more sensitive to solvent – an observation that corresponds to practical experience.

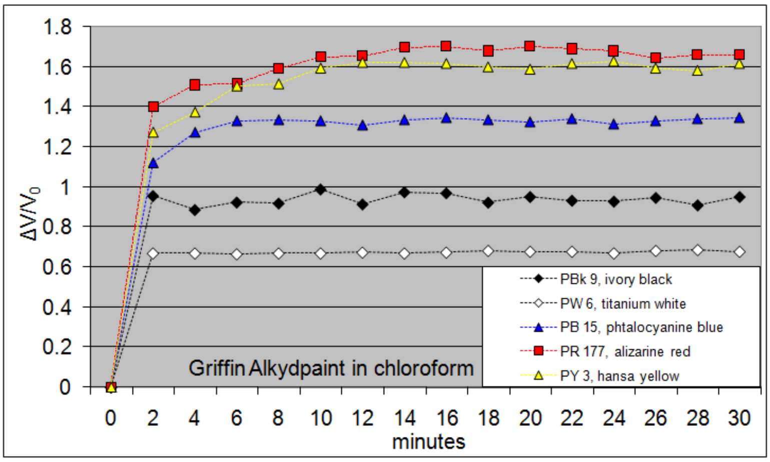

Fig. 3: Influence of the pigment on the solvent sensitivity.

Not only the extent of the swelling, but also the speed of the swelling is an important parameter. For the work of an art restorer, the increase in volume reached after one hour is merely an abstract figure, but knowing the swelling rate and the retention time could be of use in choosing the right solvent. In the 1970s, it was assumed that solvents with a high vapour pressure (like acetone and diethylether) evaporate quickly and therefore have little time to swell the paint and cause damage [Ruhemann 1968]. However, Masschelein-Kleiner [1981] established that vapour pressure and retention time are not coherent. Ethanol, for example, has an average vapour pressure but a very high retention time. This is due to the fact that the vapour pressure only plays a role on the film surface. Inside the film, interactions occur between the solvent and the paint film components that govern the retention time of the solvent. Indeed the present test results contradict the assumption of the 1970s. Solvents with a high vapour pressure (like acetone and diethylether) are not harmless when it comes to the swelling rate, swelling volume and embrittlement.

The present experiments showed that the volume increases very rapidly in the first few minutes. The DIY alkyd paints swell more slowly than the Griffin alkyd paints. The two year-old oil paints swell more slowly than the six month-old oil paints. As is known from oil paints [Feller et al. 1985, van den Berg et al. 1999], the swellability of alkyd paints also decreases with age (Fig. 4).

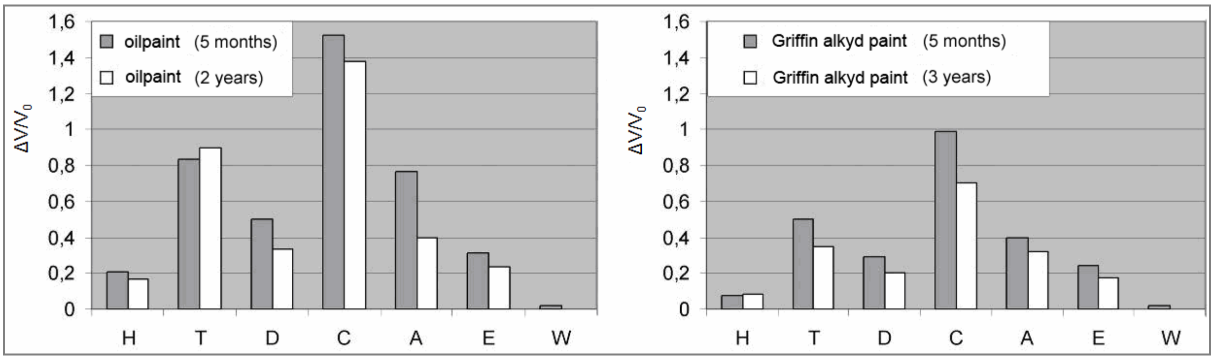

Fig. 4: Influence of the age of the paints on their susceptibility to solvent attack.
Left: Maximum swelling values of 5 month-old and two year-old oil paints in seven solvents.
Right: Maximum swelling values for 5 month-old and 3 year-old Griffin alkyd paints in seven solvents.
With both paint systems, the sensitivity to solvents decreases with age.
(H = hexane, T = toluene, D = diethylether, C = chloroform, A = acetone, E = ethanol, W = water)

With most solvents, the maximum swelling is reached after only two minutes (Fig. 5). Water, on the other hand, produces only a very gradual increase in volume, which has still not been concluded even after 30 minutes. Fig. 5 shows the swelling profiles and the maximum volume increase of the three different paints in the seven different solvents. The results for the blue-pigmented paint strips are illustrated here as an example. If we compare the three paint systems, it can be seen that all three react to the same solvents in the same way, either more sensitively or less sensitively. This means that an art restorer can basically apply the experience gained from restoring oil paintings to alkyd paints. Alkyd paints are slightly less sensitive to swelling than oil paints.

Chloroform, because of its very good dispersive interaction ability, shows by far the greatest swelling potential in all cases. Acetone and toluene also exhibit significant swelling. Towards the outer edges of the polarity scale, the ability of the solvents to interact with the paints – and thus their ability to swell – constantly decreases.

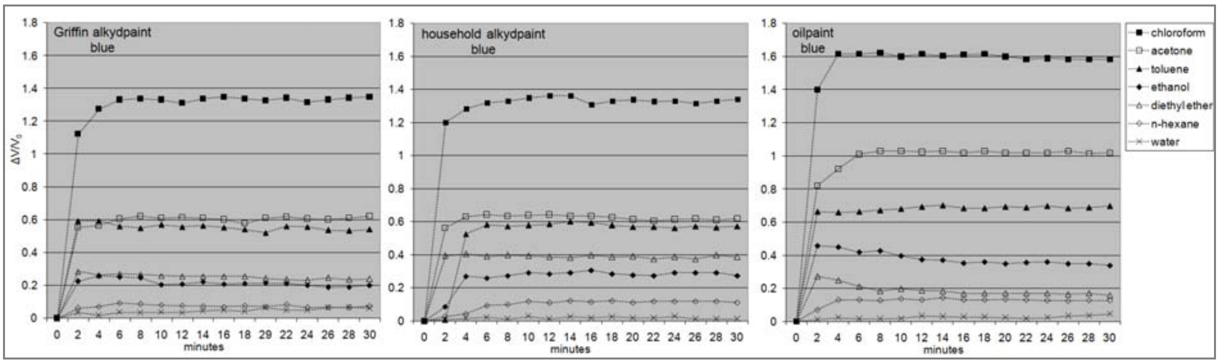

Fig. 5: Swelling rate and maximum volume increase in seven different solvents.
Left: Griffin alkyd paint, centre: DIY alkyd paint, right: oil paint (paint strip 5 months old).

Leaching: Analysis of Solvent Extractable Components

This chapter deals with the extractability of binder components and low-molecular weight decomposition products. The phenomenon of leaching in oil painting was examined back in the 1930s [Phenix et al. 2001]. For the work of an art restorer, leaching is a more difficult problem to understand than the swelling of the paint. Swell tests are also easy to carry out in practice and can help with the choice of a suitable solvent. On the other hand, the leaching out of low-molecular weight components is not visible during the practical work, and its analysis requires more complicated equipment. Since leaching is a hidden risk that has permanent consequences for an oil painting, extensive studies have been carried out on the subject [Feller et al. 1985, Mills et al. 1979, Sutherland 2001]. On the other hand, no publications have yet appeared (to the best of our knowledge) on the subject of leaching with alkyd paints.

Extracts from the white Griffin alkyds and the white DIY alkyds were obtained with the solvents n-hexane, toluene, diethylether, chloroform, acetone, ethanol and water and analysed using FTIR[3]. Fig. 6 shows the infrared spectra of the seven solvent extracts from the two alkyd paints. On the left are the infrared spectra of the extracts from the Griffin alkyd, and on the right, those from the DIY alkyd. The solvents are arranged in order of increasing polarity. The extracted components are various fatty acids and oxidised hydrocarbon compounds. As expected, the polarity of the extracts increases with the polarity of the solvents. The quantity of extracted substances, on the other hand, does not increase with the polarity of the solvent. The quantity is largest with chloroform and acetone and declines considerably at both ends of the polarity scale. The components extracted from the DIY alkyd paints are basically similar to those from the Griffin alkyds. The same components are leached out. Like the infrared spectra of the dried paints, the DIY alkyds have a higher proportion of aromatic polyester structures.

The results of the infrared spectroscopy analysis largely conform to the tests carried out by Sutherland et al. [1999, 2001] on oil paints. He described solvent extracts from test paints and also from painting samples from different dates. Here, he also found free fatty acids, oxidised low-molecular weight fatty acids and higher-molecular weight mono-, di- and triglycerides. Prolonged exposure to solvents also encourages the leaching out of higher-molecular weight components from the partially crosslinked material. Applying this to the practical work of an art restorer, Sutherland recommends that the period of exposure to the solvent should be as short as possible. If a painting is revarnished after varnish removal, the paint should be given time to dry before application of the new varnish. Erhardt and Tsang [1990] and Tumosa et al. [1999], too, detected saturated fatty acids and polar dicarboxylic acids. Because of the analogous degradation processes of alkyd paints and oil paints, the same decomposition products are to be expected. Accordingly, the paint systems show comparable behaviour in the extraction of such components.

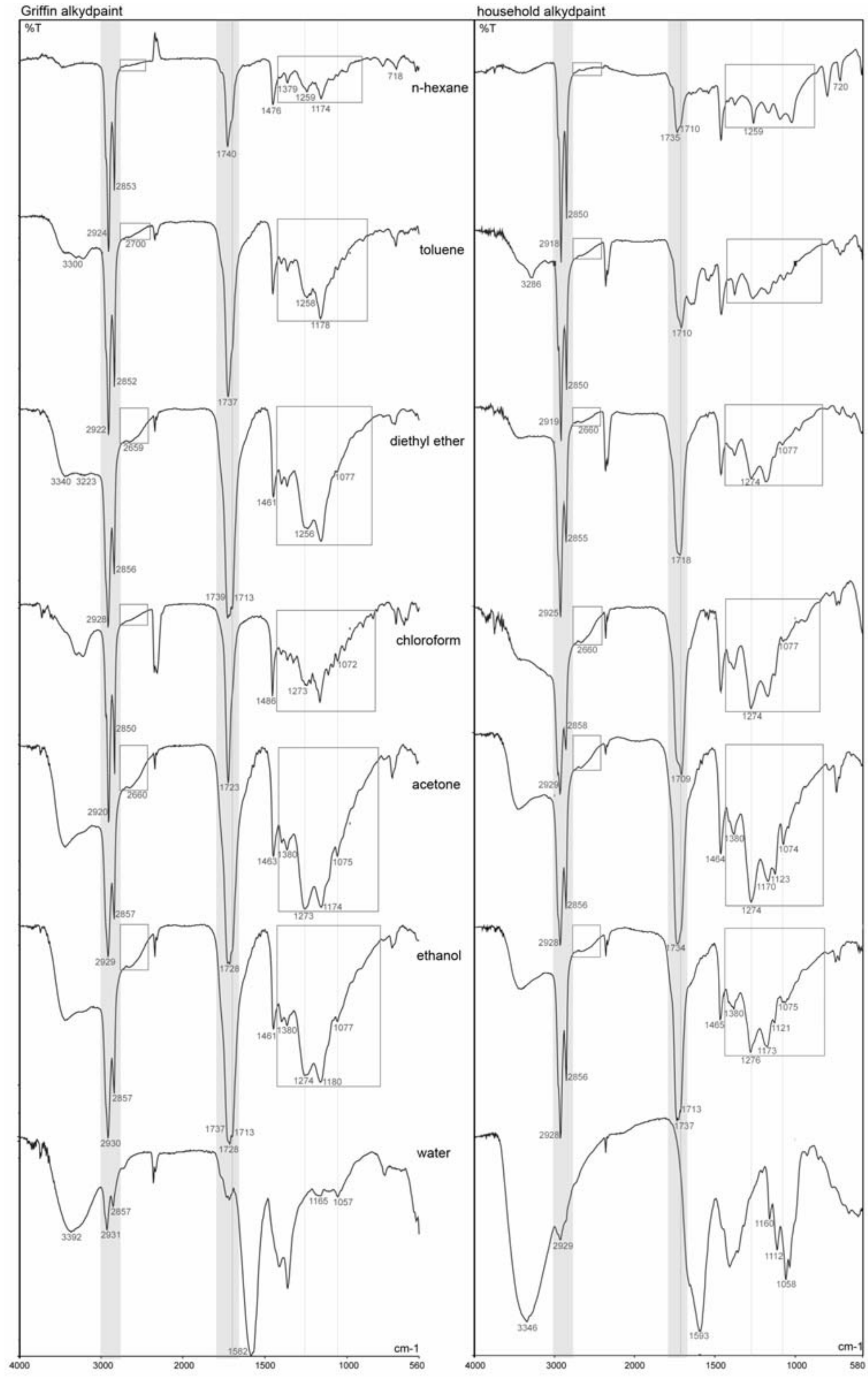

Fig. 6: Infrared spectra of the solvent extracts. The polarity of the solvents increases from top to bottom. Left: extracts from the white Griffin alkyd paint, right: extracts from the white DIY alkyd paint.

Stress-Strain Measurements: Mechanical Behaviour and Embrittlement through Exposure to Seven Different Solvents

Research on the damage to the paint layer caused by solvents normally concentrates on the previously described phenomena of swelling and leaching. Studies on changes to the physical and mechanical properties due to treatment with solvents are only rare. The swelling of the paint layer occurs directly during the solvent treatment. Leaching is an accompanying side-effect that can change the physical properties. This means irreversible damage to the structure of the painting and it is this, in fact, that is the real danger of solvent application. This paper attempts to establish the extent of the long-term damage by plotting the influence of the solvents on the elasticity modulus of the oil paints and alkyd paints. To establish the influence of the pigments on the mechanical properties, black, white, blue, red and yellow paint strips were examined. The paint strips were immersed for one minute in n-hexane, toluene, diethylether, chloroform, acetone, ethanol and water. After drying, the length of the strips had decreased significantly – in the case of the strips treated with acetone, by as much as 6.7% compared with their original length. The extent of the loss of volume cannot be explained just by leaching. The density of the film evidently increases. As now, Erhardt and Tsang [1990] established significantly more pronounced shrinkage in the case of the strips treated with acetone than with those treated with ethanol, even though the quantity of leached components was the same. The tensile tests produced meaningful results in several respects. On the one hand, it becomes clear how different the three different paint systems are in terms of their extensibility and break resistance. Fig. 7 shows as an example the stress-strain curve of the untreated blue paint strip. The DIY paints showed by far the highest tensile strength in all cases and the lowest extensibility. The Griffin alkyd paint and the oil paint had very similar mechanical properties. Although the two artists' paints have a lower tensile strength, they are very much more extensible than the other paints.

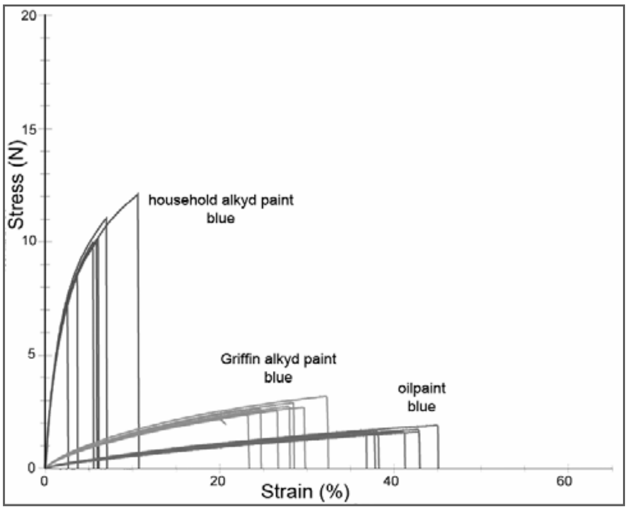

Fig. 7: Stress-strain diagram of the blue pigmented untreated DIY alkyd paints, Griffin alkyd paints and oil paints.

The paints differ considerably from one another because of the various additives and fillers. The same applies to the differing pigmentation, which has a dominant effect on the elastoplastic behaviour of the paint film (Fig. 8).

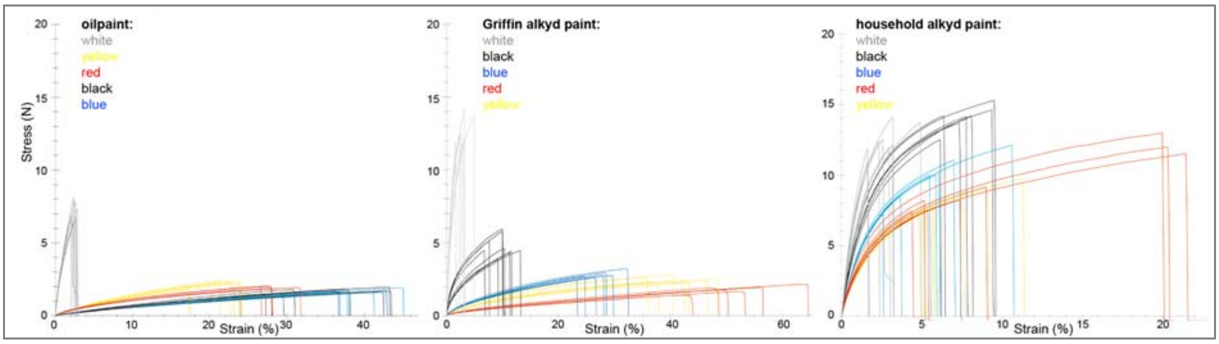

Fig. 8: Stress-strain diagram of the untreated paints. Influence of the pigmentation on the mechanical properties of the untreated oil paint (left), Griffin alkyd paint (centre) and DIY alkyd paint (right).

After only one minute's immersion in solvent, the properties of some of the paint films change significantly. Fig. 9 shows as an example the results of the yellow oil paint, Griffin alkyd paint and DIY alkyd paint. Irreversible embrittlement has occurred with all the solvent-treated paint films. Only the paint strips immersed in water do not exhibit any significant change in their mechanical properties, compared to the untreated reference paint strip. A lasting softening effect of the water, as observed by Hedley et al. [1990], was not found. Among the seven solvents, the highest level of embrittlement was expected to come from the chloroform, which caused the greatest swelling and, in quantitative terms, has the highest leaching potential. However, it was acetone that caused the heaviest embrittlement with all the paint systems and all the pigmentations (Fig. 9). This was surprising in view of acetone's moderate swelling effect. The increase in brittleness is, however, coherent with the shortening in the length of the strips, which was ascertained after evaporation of the solvent. It is possible that the morphology changes, and a denser film is formed, as Hedley et al. [1990] and Feller et al. [1985] suspected. The heavily swelling chloroform also leads to substantial permanent changes in the mechanical film properties. As with the swelling behaviour, the damage declines towards the edges of the polarity scale. Consequently, n-hexane and water barely have any effect on the mechanical properties of the paints. On the other hand, diethylether, which has relatively low swelling potential and only results in the leaching out of small quantities, exhibits relatively pronounced embrittlement. Like the paint strips treated with acetone, those treated with diethylether also shrink considerably in length. If the properties of ethanol are compared with those of acetone, it is surprising to note the minimal influence of the ethanol on the mechanical properties of the paint films, because the analysis of the extracts showed that the components and quantities leached out from the Griffin and DIY alkyd paints by ethanol and acetone are comparable.

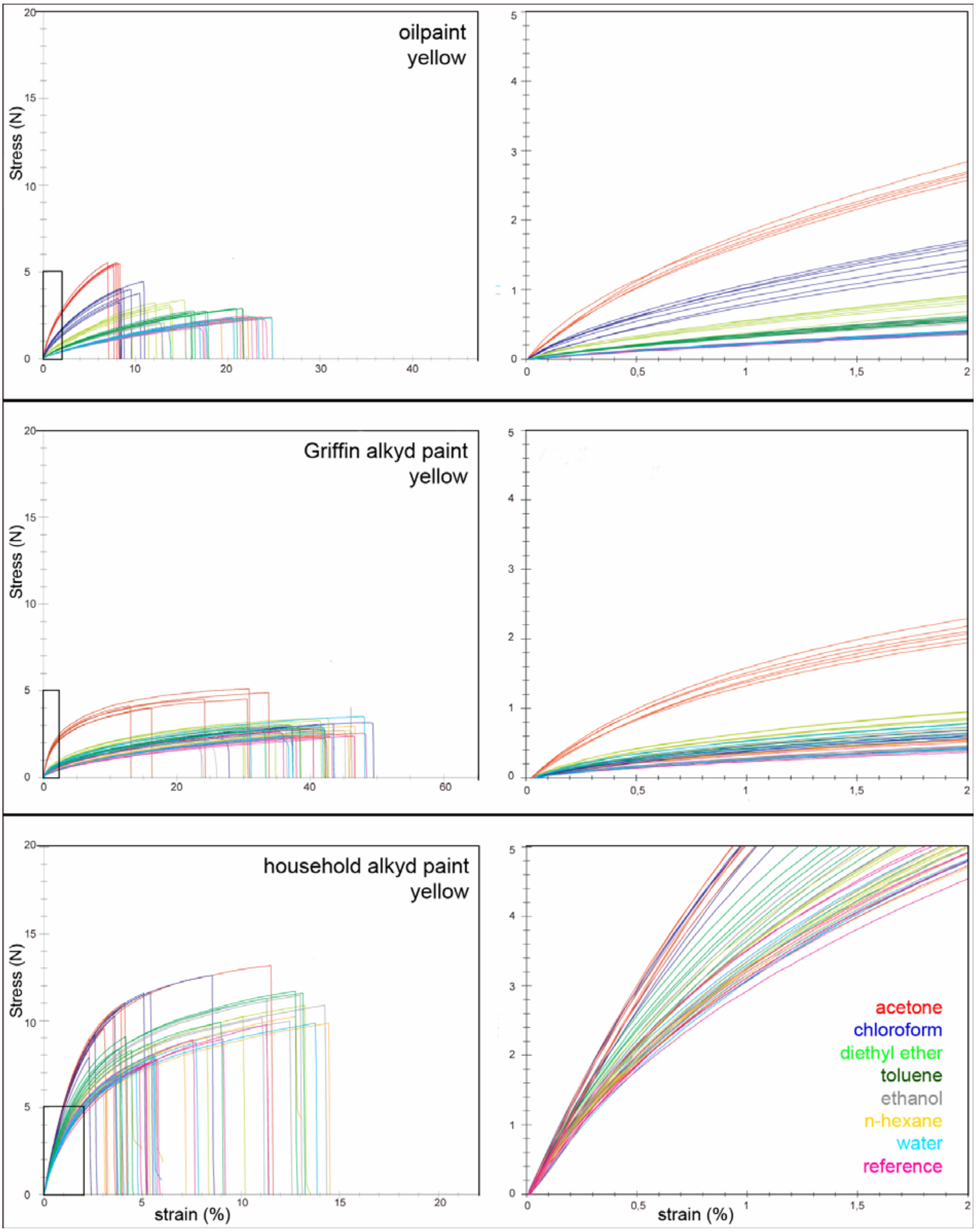

Fig. 9: Influence of solvent treatment on the mechanical film properties of the yellow oil paint (top), Griffin alkyd paint (centre) and DIY alkyd paint (below). Left: Total stress-strain diagram. Right: Detail of the elasticity modulus at 5N tensile force and 2 % elongation. Acetone causes the most marked embrittlement of the paint strips, followed by chloroform, diethylether, toluene and ethanol. N-hexane and water barely alter the mechanical properties at all.

DISCUSSION AND CONCLUSIONS

The present study shows that alkyd paints are related to the customary drying oils not only in their drying mechanisms but also in their technical properties. Household alkyd paints and artists' alkyd paints react to the same solvents as modern oil paints, whereby alkyd paints as a whole have somewhat higher solvent resistance. Experience gained in the use of solvents on oil paintings can be largely applied to alkyd paints.

The results of solvent susceptibility basically reinforce to what has been reported so far in the relevant literature: Through the swelling of the paints, leaching is made possible. The leached-out components have a softening character. If such components are missing, the inevitable result will be irreversible embrittlement [Feller et al. 1985, Hedley et al. 1990, McGlinchey 1991, Tumosa et al. 1999, Erhardt et al. 2005]. It was, however, found that this generalisation should be specified, because particularly the results of the tensile tests contradicted the results put forward by the theories in literature. The swelling potential and leaching tendency cannot be regarded as the only criteria for solvent sensitivity because these factors do not correlate with the mechanical changes of the material. The swelling potential is unarguably an important measure of the sensitivity to solvents. If the original paint layer swells, for example, during varnish removal, it can easily become damaged through the mechanical stress of the cotton wool pad. This process naturally also has an influence on the transport of solvable molecules. However, there is no absolute dependence with regard to the amount of leached material, which is due to the polarity difference between the low-molecular weight degradation products and the matrix system. These differing solvation effects thus determine the effect on the entire system. In literature, it is assumed that the leaching out of these low-molecular weight components results in embrittlement of the paint layer. This theory is confirmed to a certain extent in as far as solvents like n-hexane and water, which produce only slight swelling and have limited dissolving properties, cause very little structural damage to the solute. These tests with solvents from different solvent categories furnish evidence that morphological structural changes are probably responsible for the mechanical changes to the material. What is meant by this is heterogeneous accumulations and depletions of various components within the paint layer. Hedley et al. [1990] indicated that the morphology of the paint layer can be influenced by the swelling and leaching and that, as a result, the mechanical and physical properties can change. The striking point is that solvents with a very high vapour pressure cause severe material changes. Similar phenomena were observed with emulsion paints, which would lead to the conclusion that the physical properties of the solvents could be the determining factor [Zumbühl 2008]. The embrittlement observed on the alkyd and oil paints cannot be comprehensively explained on the basis of the present data, but the influence of the various factors can be defined. Future research will focus on examining the diffusion rate and retention time of the solvents and analysing the morphological structural changes.

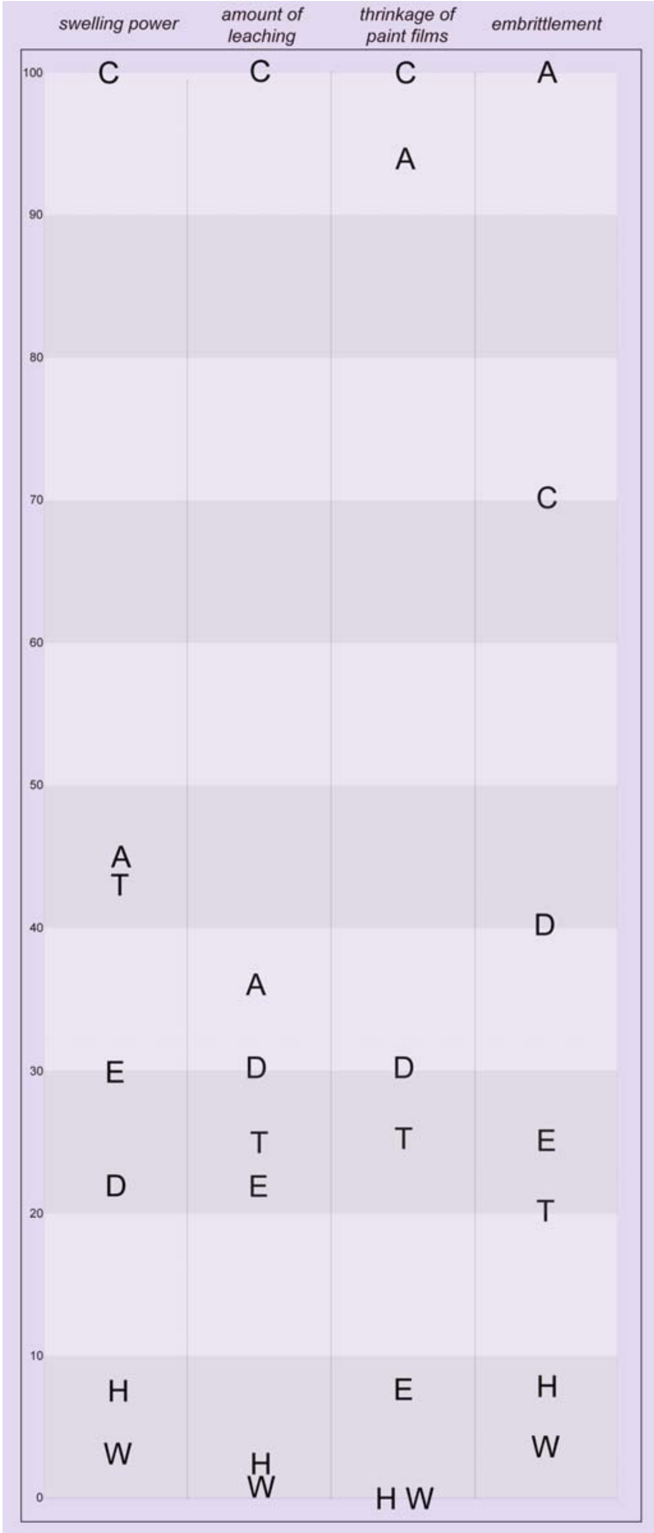

Fig. 10: Comparison of all the test results: They are not absolute figures but relative averages. The effect of the "strongest" solvent has been put at "100", and the effects of the other solvents rated accordingly lower. The embrittlement data were optically estimated on the basis of the determined elasticity moduli. (C = chloroform, A = acetone, T = toluene, E = ethanol, D = diethylether, H = hexane, W = water)

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