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Two case studies of unsaturated polyester composite art objects

Full paper

Ulla Knuutinen[1*] and Paivi Kyllonen[2]

1. EVTEK Institute of Art and Design, Conservation Department, Lummetie 2b, 01300 Vantaa, Finland
2. Oulu City Art Museum, Kasarmintie 7, B.P 4490015, Oulu, Finland

*Corresponding author: ulla.knuutinen@evtek.fi

Abstract

Three dimensional art objects and parts of installations made of composite materials containing gypsum, fibreglass and two-component unsaturated polyester (UP) resins in the collections of Oulu City Art Museum, Finland, were studied. Even if composite materials are considered to be chemically resistant, the artefacts made of UP composites have started to undergo degradation processes.

The aim of this pilot project was to study the causes of degradation of the composite materials. Ageing and degradation tests and analyses were employed to reveal their effect on unsaturated polyesters. To determine the chemical composition and colour changes of the tested materials, samples were analysed before and after ageing using FTIR and CIE L*a*b* colour measurements.

Analyses of this preliminary study have shown that there are differences in quality, curing and ageing properties of different UP composites. This information was found to be important in developing conservation strategies for art objects made of UP composites.

1. Introduction

Under comparable conditions, ageing of the majority of synthetic materials is known to proceed faster than ageing of traditional materials. Objects made of plastics may degrade very rapidly and can have a useful lifetime of only a few decades. At the same time, modern materials (from cellulose derivates to synthetic polymers) have become increasingly important in art collections, because, since the earliest production of synthetic materials, artists have explored their use as sculpturing materials.

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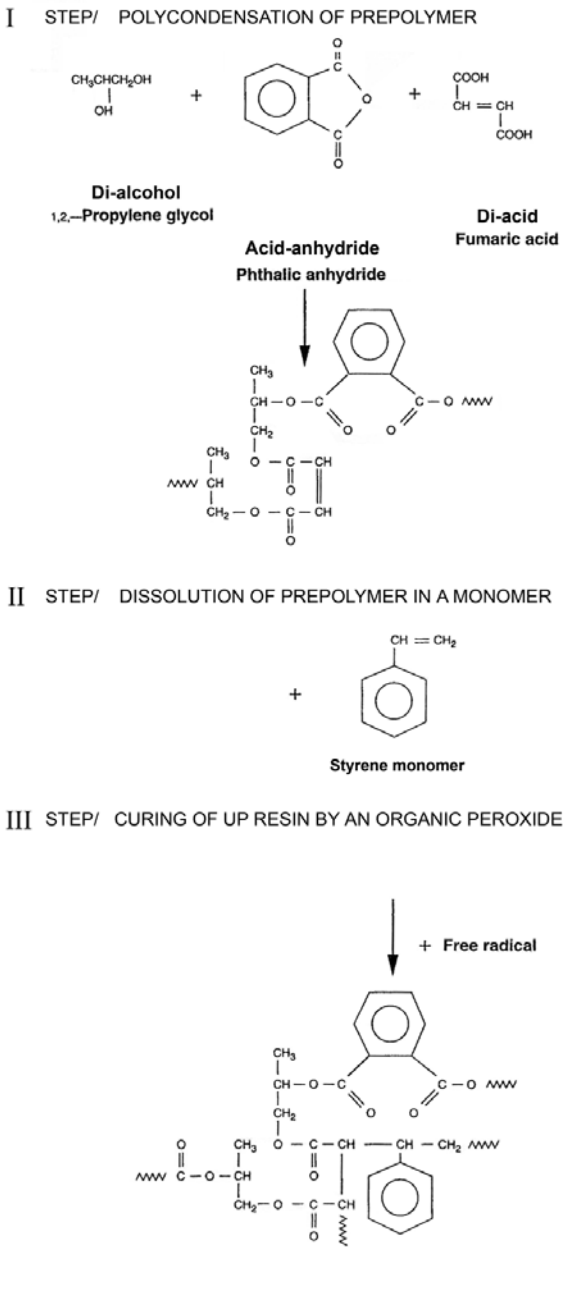
In museum collections, objects and artefacts made of unsaturated polyester resins (UP resins) reinforced with fibreglass are relatively new compared to other synthetic materials. There is insufficient information available on analytical research and conservation strategies for unsaturated polyester materials.[1-3] When unsaturated polyesters are used as part of composite materials with gypsum and fibreglass, they are considered stable with good chemical resistance and weathering qualities.[4,5] However, prolonged exposure to ultraviolet light as well as changes in temperature and humidity can cause chemical and physical degradation of unsaturated polyesters.[6] This is especially true of artefacts made of unsaturated polyester resin reinforced with fibreglass exhibited in the open-air.

Polyesters are generally regarded as stabile polymers, and are reaction products of etherification of di- or poly-hydric alcohols with di- and poly-basic acids and anhydrides. They fall into two broad categories: saturated polyesters and unsaturated polyesters.[7]

Polyesters, which are used in composite materials in combination with fibreglass reinforcement, belong among unsaturated polyesters (UP resins). They are formed in three steps (Figure 1). First, the polycondensation reaction of a di-alcohol, an acid anhydride and a di-acid into a pre-polymer resin; second, addition of a monomer; and third, curing of the polyester resin into a thermoset polymer by an organic peroxide. The unsaturated di-acid or acid anhydride allows molecules to crosslink the chains by formation of bridges. In a thermoset UP polymer, the macromolecules are cross-linked, forming a rigid, three-dimensional network structure. Once these cross-links are formed during the polymer reaction, the thermoset polymer cannot be melted and reshaped by the application of heat or pressure.[5,8,9]

In practice, the processes are much more complex, especially in commercial products where several different alcohols and unsaturated acids may be used in the preparation of polyesters and many different monomers can be used as diluents. If a resin component contains styrene as a solvent it also copolymerizes with the unsaturated polyester. Styrene is the most widely used diluent because it has low viscosity, because it is a good solvent and because of its low cost. Its drawbacks are flammability and health hazards. The addition of styrene can be as high as 50% depending on the product. Unsaturated polyesters can further be cured following a free radical mechanism that can be controlled using various chemical additives: initiators, accelerators and inhibitors.[9]

Unsaturated polyesters are widely used as chemically resistant finishes for interiors of chemical and petrochemical storage tanks, and as coatings for boats and bathroom fixtures.[9] In UP composites, unsaturated polyesters are used in combination with fibreglass in order to produce strong, rigid structures. In artworks, it has been popular to use an unsaturated polyester resin to create a strong three-dimensional shape of fibreglass-reinforced



Figrue 1: Scheme of synthesis and curing reactions of UP resin.

polyester. A fibreglass mat, dipped in fluid resin, can be used to lay up piece by piece, to cover a shape cut from foam, or to lie out in a mould.[3,5,10,11]

The Oulu City Art Museum houses a collection of artefacts made of unsaturated polyester composite materials, which have visibly started to undergo deterioration. From the collection, two artefacts were selected for this preliminary study of modern polyester composite materials. In Figure 2 we show the installation “The Last Milk Platform”, from 1992, by Jan-Erik Andersson, which is constructed as a wooden white, black and blue milk platform with an aluminium milk churn and a red reinforced

Figure 2: “The last milk platform”, 1992, by Jan Erik Andersson.

Figure 3: “Cocotte with two dogs”, 1987, by Kari Tykkylainen.

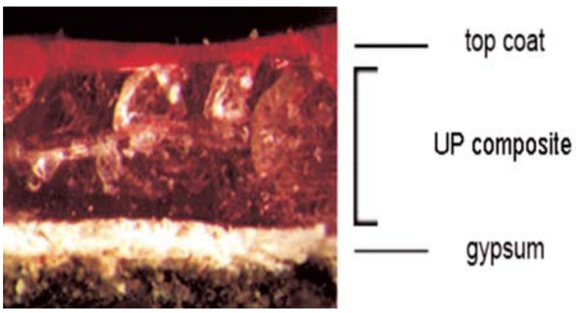


Figure 4: Cross-section of the mermaid (cf. Fig. 1) UP composite layers.

polyester mermaid on its roof. A metal stick is fastened to the milk churn pointing out with a black-white bat and a yellow ball. This installation was exhibited outdoors. Another artefact chosen from among installations stored indoors was “Cocotte with Two Dogs” (1987) by Kari Tykkylainen, which consists of a three-dimensional black female figure and two dark red-brown dogs with metal chains (Figure 3).

In order to study the causes of degradation and to propose conservation procedures for artefacts made of unsaturated polyester resins, a pilot project was carried out in the conservation department of the EVTEK Institute of Art and Design in cooperation with the Oulu City Art Museum.

2. Materials

2.1 Material description

Figure 4. we show the layered structure of UP composite found in the mermaid sculpture, part of the artwork shown on Figure 1. Covering the gypsum, there is a layer (or layers) of UP resin composite containing a mixture of UP resin and fibreglass. The uppermost layer, which is very often coloured, is called the top coat or the gel coat. The gel coat (free of fibreglass) is a layer spread in negative moulds before the fibreglass mat or tissue is impregnated with resin. In hand lay up models, the uppermost layer is usually called the top coat.[11] The layer structure of unsaturated polyester composite art objects can also be different than the structure presented in Figure 4, as some layers may be missing or could be incomplete.

2.2 Artist interviews and documentation

The artists who had used composite materials were interviewed and asked to give step-by-step and detailed structural and material information about their artefacts. Art objects were also documented in the Oulu City Art Museum. The shape of the mermaid on Andersson´s installation (Figure 2), which was made of plaster of Paris bandages (Gypsona®, calcium sulphate attached to cotton cloth), appeared to serve the demands of sculpturing a human figure. The polyurethane foam was extruded into the inside of the cast figure skin and some polystyrene, wood and metal particles were added. Then unsaturated polyester resin, as a composite with fibreglass, was used over the hand lay-up mould and covered with a pigmented polyester topcoat[12].

The Installation “The Last Milk Platform” by Jan Erik Adersson has been exhibited or stored from the year 1992 periodically both inside and outside: Outside in Sotkamo 6/1992-11/1993 and in Oulu 6/1995-6/2003 and inside 11/1993-6/1995 and 6/2003-present day.

The environment around the object has varied greatly. The temperature in the Oulu area in winter has fluctuated from -37.5 °C to +9.1 °C, and in the summer from -9 °C to +32 °C. The average relative humidity varied from 73% to 88% RH in winter, and in summer from 55% to 89% RH. These figures were recorded in the Climatological statistics of Finland (Finnish Meteorological Institute).

From 1991 to 2006, the temperature inside the museum fluctuated around +21°C ± 3 °C, and relative humidity around 30 - 45% RH in winter, and 45 - 60% RH in summer.

Tykkylainen´s installation (Figure 3) was formed using two methods: the female figure was made of iron wire net, covered with plaster of Paris as a cast, with an addition of textile material in-between. The dogs have been constructed as hollow forms of reinforced unsaturated polyester, open on the front side. The artist used self-made pigmented paint over the backs of the dogs. The coating layer was uneven and did not cover all of the back surfaces. The artist also revealed in an interview that an extra final layer of transparent acrylic paint over pigmented areas was used for UV protection.

The “Cocotte with Two Dogs” was made by the artist outside in summertime and was first exhibited outside until the end of 1990 and was then moved inside into the Oulu City Art Museum, were it has been for the past 15 years.

Both artists, Andersson and Tykkylainen used reinforced unsaturated polyester resins because they were inexpensive, easy to use on hand-made laminated surfaces and reinforced polyesters and because they form light, strong and rigid structures.

2.3 Reference samples

Three different commercial UP resin products (Table 1) were tested. The choice of commercial unsaturated polyester products and other materials and samples for testing was made on the basis of the information collected from the art objects and the literature.[5,7,9,12] The UP sample materials, three different two-component unsaturated polyester products (A, B and C) were selected so that one of them, product C, contained a UV stabilizer and other products: A and B, represented basic, inexpensive UP products, which artists are known to have used in their works of art in Finland. The product information of the tested commercial UP resins is given in Table 1.

Product	Name	Company
A	Synolite	Bang & Bonsomer
B	Norpol 540-800	Terpol Oy
C	Norpol 450-500	Terpol Oy

Table 1: Product information regarding the commercial UP resins used as reference samples.

Glass was used as a ground material for sample preparation. Samples of four different structures were prepared from each polyester material: the first containing only unsaturated polyester, the second contained UP resin with gypsum (Gypsona®, Smith Nephew Medical Ltd), the third included fibre glass (Ahlstrom M 501, 300 g/ m^2) and UP resin together, and the fourth with all layers found in artefacts: gypsum, unsaturated polyester and fibre glass. After the preparation, the samples were left to dry and cure for several weeks to ensure complete dryness.

3. Methods

3.1 Accelerated degradation

Accelerated degradation was performed using the QUV instrument (Q-Panel Company) by cycling temperature, relative humidity and irradiation with UV-light.[13] The QUV Instrument was programmed to follow repeated 4-h cycles: first, the temperature was kept at 60 °C with application of UVB light, then continuing for another 4 h at 40 °C and 100% RH. The samples were kept in the ageing chamber for 4 weeks. The protocol of weathering was based on the SFS-EN- ISO 11507:2001 standard for testing of paints and varnishes, exposure of coatings to artificial weathering.[14]

3.2 Microscopy

Stereo-microscopy was used (Wild 3 M B, magnifications from 1,6x to 40x) to investigate the artefacts and the reference samples in detail.

3.3 Colourimetry

Colourimetry was performed using a Minolta CM 2600d colourimeter. The CIE L*a*b* colour space (also referred to as CIE LAB) was used with a 10° Supplementary standard observer to measure colour changes in samples before and after QUV ageing.[15,16] Colour measurements were made according to the ISO 7724-1 standard, which describes the fundamental requirements for determination of colour co-ordinates of paint films and related materials.[17] All CIE L*a*b* parameters were determined, but the most useful parameter to describe colour changes, the colour difference was parameter b*, which is a measure of yellowness. Five readings in different locations were taken on each sample to take account of the heterogeneous nature of surfaces and in order to count the mean value and standard deviation of each measured area.

3.4 FTIR analyses

To characterise chemical changes in the material during degradation, characteristic Absorption /

transmission IR bands of unsaturated polyester resins made from commercial UP products and artefact samples were studied.[18,19] FTIR analyses were performed using the Nicolet Impact 400 IR. The results were processed using Omnic version 5.1 software (Nicolet Instrument Corporation). In cooperation with Tikkurila Oy , FTIR microscopy was performed using a Perkin-Elmer One FTIR I-Series microscope.

5. Results and discussion

Using a stereomicroscope (Figure 5), it was possible to see both small cracks and particles of soiling, especially on the porous surface area of the mermaid. The transparent polyester resin between the cracks appeared yellow. The reason for cracking are different shrinkages of the UP composite layer and the top-coat layer. Through the pores and along the glass fibres, water may migrate into the unsaturated polyester layers. Air inclusions in the polyester composite layer and between the top-coat and composite layer were also found. It appeared that water might have migrated into the pockets of air and caused damage while freezing.

In studies of commercially available UP samples (Table 1), stereomicroscopy provided an indication of different curing and ageing properties of the different resin products. A significant number of air

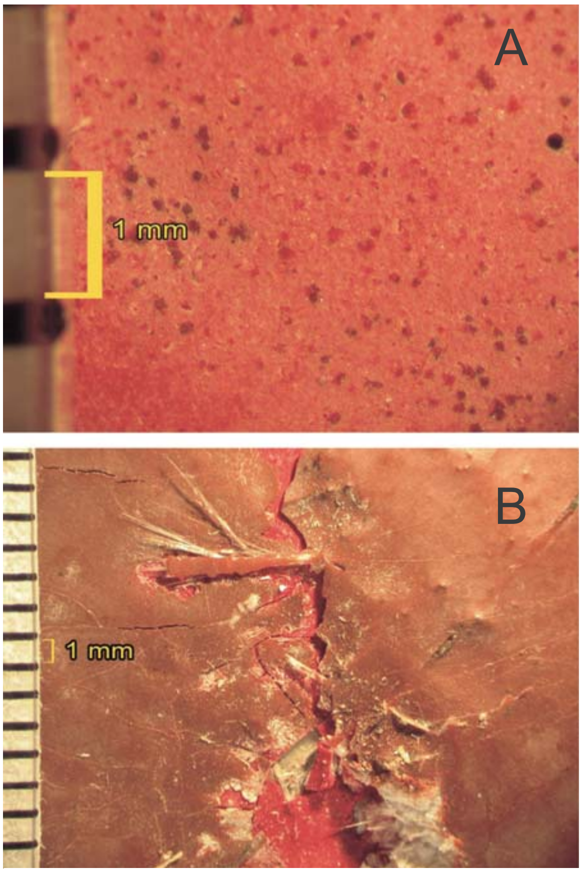


Figure 5: Stereomicroscope microphotographs of the UP material from the mermaid (Figure 1). A: porous area, and B: formation of cracks on the surface.

inclusions in unsaturated polyesters A and B was found after curing (before QUV ageing). Furthermore, cracks developed on these two products during the ageing process. The product C containing a UV stabilizer had best curing properties and it proved to be the most stable: a limited number of air inclusions formed after curing, and polyester remained uncoloured throughout the artificial ageing.

The same indications of damage, air inclusions, pores and cracks in the UP gel coat and in fibreglass reinforced polyester resin, have also been reported during the AXA Art Conservation Project [2].

5.1 Colour Measurements

From the colour measurement results, Table 2 shows colour changes/ changes in the yellowness (*b**) of pure polyester film samples, and also in samples where polyesters were used as a part of the composite, unsaturated polyester with fibreglass. The colour measurement results are presented as b^* values and Δb^* values under Standard illuminant D-65 (Daylight 65). From the CIE $L^*a^*b^*$ parameters, b^* values were chosen to present colour measurement results, because the most significant changes were observed in the parameter b^* (yellowness).

Sample	b* before ageing	b* after ageing	Δb*
A (pure polyester)	2.6 ± 0.1	6.2 ± 0.4	3.6
B (pure polyester)	5.6 ± 0.4	12.1 ± 0.5	6.6
C (pure polyester)	1.3 ± 0.1	4.2 ± 0.4	2.9
A (composite)	9.6 ± 0.5	17.0 ± 0.2	7.4
B (composite)	21.7 ± 0.4	26.0 ±0.6	4.3
C (composite)	7.8 ± 0.2	13.2 ± 0.4	5.5

Table 2: Colour changes of UP products: samples A, B and C and their composites (UP resin + fibreglass) presented as b* before and after QUV ageing.

Among the pure unsaturated polyester samples, the smallest change was found in UV stabilized product C with Δb^* value 2.9, and the biggest change in product B with Δb^* 6.5. The products A and B already appeared yellowish after curing before the artificial ageing was begun. The uncertainty of determination of b*, expressed as standard deviation (S.D.) varied from 0.1 (sample C) to 0.5 (sample B). The differences in S.D. may indicate the quality of products and could be linked to the curing process of the UP resins: the smaller the uncertainty, the more even the curing process. During the QUV ageing the samples also became more heterogeneous.

The polyesters seemed to exhibit different extents of yellowing when tested individually or when studied as a component of the composite. Gypsum in combination with polyester did not seem to have a drastic effect; but polyesters containing fibreglass and the composite of gypsum, fibreglass and polyester exhibited the most significant colour changes. The product B exhibited the most signifi-

Figure 6: The yellowed UP composite layers from the dogs of the installation by Tykkylainen (Fig. 2).

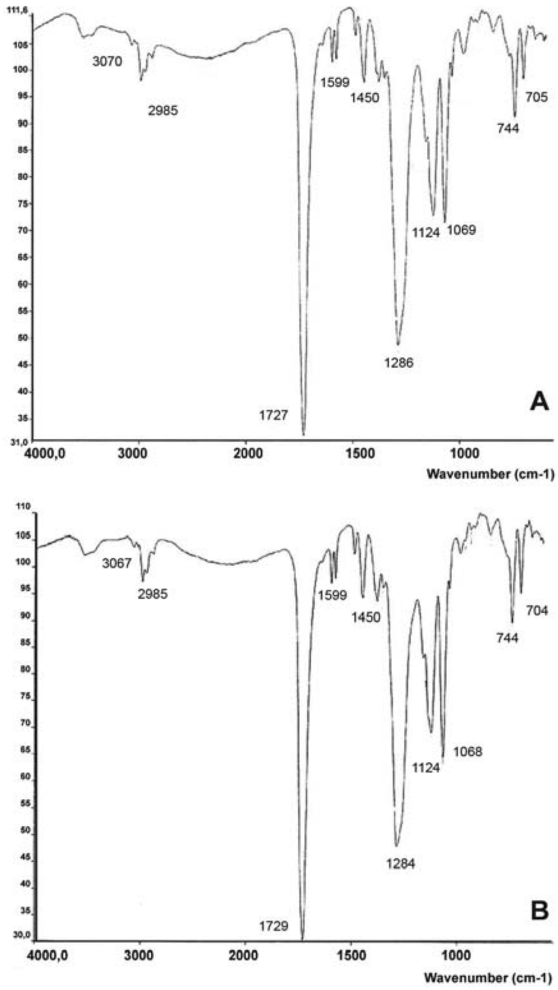


Figure 7: FTIR transmission spectra of the UP product C. A: before, and B: after QUV ageing.

cant yellowing (b* = 21.7) already after curing (before artificial ageing). This indicates that fibreglass affects the curing process of UP composite. This effect deserves a further in-depth study.

From the uncertainties, similar conclusions can be made regarding inhomogeneity of the composite samples as for pure unsaturated polyester samples. The most inhomogeneous sample after QUV ageing was the product B with S.D. = 0.6.

It was possible to determine the yellowing indices directly from artefact surfaces, because there were areas with no protective paint layers, no top coat, and the fibre glass reinforced unsaturated polyester was exposed to light as well as to changing temperature and humidity. Figure 6 shows a detail of the dog in Tykkylainen's installation, where exposed UP composite layers and UP resin with fibreglass are evident. These areas were strongly yellow b* varied from 13.7 to 17.7.

5.2 FTIR Spectroscopy

The resistance to weathering properties of the UV stabilized product C were also verified using FTIR. In Figure 7, we show the FTIR transmission spectra of product C before and after ageing. In both cases, it was possible to find characteristic bands of unsaturated polyester resins:[18,20,21] both unsaturated C=H stretching vibrations at 2985 cm^{-1} and saturated, aliphatic hydrocarbon C-H stretching between 2985 cm^{-1} and 2880 cm^{-1}, two aromatic, ring 'breathing' bands: a doublet at 1600 cm^{-1} and an additional peak at 1450 cm^{-1} , unsaturated in-plane deformation at 1070 cm^{-1}, unsaturated, aromatic out-of-plane bending deformations at 744 cm^{-1} and 705 cm^{-1}. In UP resins, there can be two or more different sources of aromatic moieties: usually from styrene and phthalic acid anhydride. Most characteristic of the FTIR spectra of UP resins is the strong carbonyl stretching between 1725-1730 cm^{-1}, and two other bands characteristic for oxygen containing groups at 1285 cm^{-1} and 1125 cm^{-1}.

Table 3 lists the frequencies of the salient peaks of the tested UP resin products before and after QUV ageing, and also contains two UP composite samples from the mermaid. The wave number values show that there is a shift of a carbonyl stretching group when the spectra information of the UP resin product is compared before and after QUV ageing. The biggest shift is 4 wave numbers in product B, from 1724 cm^{-1} to 1728 cm^{-1}; and 2 wave numbers in products A and C. Furthermore, the wave number values for these carbonyl stretching frequencies of 15-year-old artefact samples are: 1730 cm^{-1} and 1731 cm^{-1}. The shift in the carbonyl group is probably caused by photo-oxidation and cross linking, which may continue during ageing. This will increase the cross linking density and may

Sample	Wavenumber (cm^{-1})							
	v=CH and v_aCH_2 v_sCH_2	vC=O	vPh	v(C=O)O	vO-C, δ=CH	δ=CH	γ=CH	γPh
A before ageing	3070, 2983	1726	1599, 1449	1285	1124	1069	744	706
A after ageing	3071, 2984	1728	1599, 1449	1284	1124	1068	744	705
B before ageing	3070, 2982	1724	1599, 1450	1289	1124	1070	745	706
B after ageing	3070, 2983	1728	1599, 1450	1286	1124	1070	745	706
C before ageing	3070, 2985	1727	1599, 1450	1286	1124	1069	744	705
C after ageing	3070, 2985	1729	1599, 1450	1284	1124	1068	744	704
Mermaid/UP layer 1	3063, 2957	1730	1600, 1494	1285	1123	1069	745	702
Mermaid/UP layer 2	3062, 2934	1731	1600, 1493	1283	1123	1068	745	701

Table 3: Characteristic FTIR bands of the commercial UP resin products A, B and C before and after accelerated degradation and two samples from the mermaid sculpture.
Legend: δ = in-plane deformation, γ = out-of-plane deformation, v_a = antisymmetric stretching, v_s = symmetric stretching, Ph = phenyl

explain why UP resins can become more brittle with age, especially if the curing process is not carefully balanced by initiators, accelerators and inhibitors, and if the product doesn't contain any UV stabilizer.

In the tested UP resins, there is also another small shift, which can be found after QUV ageing, towards lower wave numbers in the fingerprint region of the dominant carbonyl stretching peak around 1285 cm^{-1}. The shift in UP product B is three wave numbers, from 1289 cm^{-1} to 1286 cm^{-1}.

In the inexpensive unsaturated polyester products, A and B, the FTIR spectra revealed other changes after QUV ageing. These changes were a broadening of bands in the fingerprint region at wavenumbers 1290-1000 cm^{-1}. These changes, accompanied by an increase in absorbance corresponding to ester linkages, have been associated with ageing and oxidation of ester group-containing paint binders.[19,22] In Figure 8 we show FTIR spectra of the product B before and after QUV ageing. Because of the evident chemical changes caused by the accelerated ageing process, the product B seems to be the most unstable one among the tested UP products.

Similar broadening of the bands was found in the samples taken from artefacts. As an example, Figure 9 shows the FTIR transmission spectrum of the UP composite from a cross-section of a sample from mermaid. This spectrum differs from the spectra of the tested resin products especially in the region 4000 - 2500 cm^{-1} (most evidently at 3500 cm^{-1}), which are the frequencies associated with –OH stretching. This is partly because this

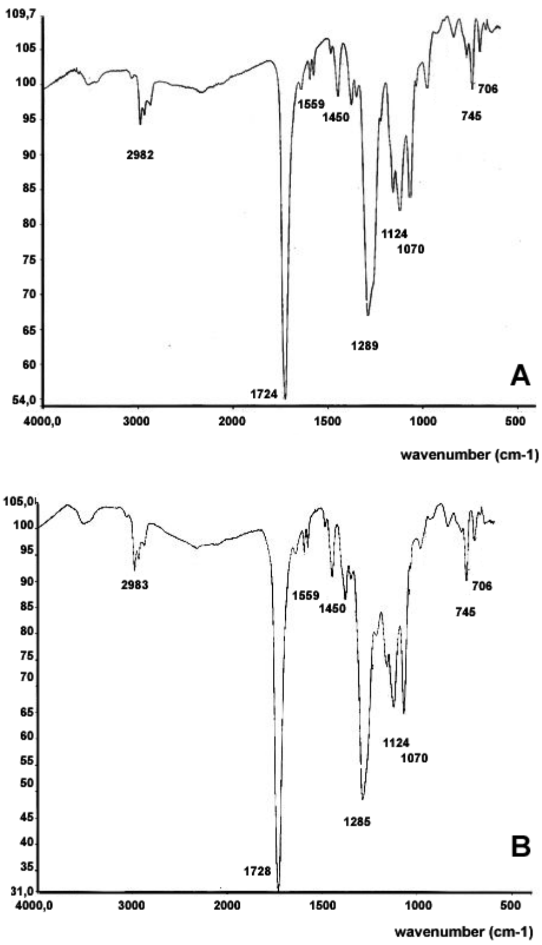


Figure 8: FTIR spectra of the UP resin B before (A) and after QUV ageing (B).

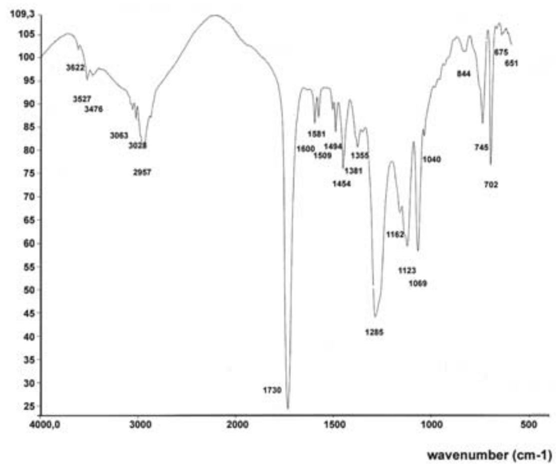


Figure 9: FTIR transmission spectra of the UP composite layer (cf. Fig. 4) from the sample cross-section taken from the mermaid.

artefact has been exhibited in the open air for years, and as a consequence, there has been almost continuous absorption of moisture into the UP resin.[12,18] However, it is also possible that ageing and oxidation products may give rise to increase absorption at wavenumbers between 3600 and 3400 cm^{-1} [22].

In addition, FTIR analyses also indicated differences in the chemical composition of unsaturated polyester resin products and samples taken from artefacts. E.g., the amount of styrene varied from sample to sample. Although we did not study the styrene content of the tested products, this feature might be important in the deterioration process. Since styrene is an unsaturated hydrocarbon, higher styrene content influences the curing process. In order to obtain a more detailed information on the chemical composition and its effect on curing, on cross-linking frequency and on deterioration mechanisms of commercial unsaturated polyester products and UP composites, further research is needed using other analytical methods such as GC-MS or NMR.

6. Conclusions

This pilot study of UP resins and UP resin reinforced fibreglass composites has provided results which will help prolong the lifetime of artefacts made of these materials. It is essential to study and understand the behaviour and degradation mechanisms and reactions of UP resins before creating guidelines and giving recommendations for dealing with art objects made of UP resins and their composites.

We have shown that there are significant differences in the stability and ageing properties of different commercial UP products. The use of inexpensive products will lead to inferior quality of the cured composite polyester resins. The samples taken from real artefacts, gave identical analytical results as the cheap, unsaturated polyester reference products.

The changes during degradation and photo-oxidation were evaluated using stereomicroscopy, colourimetry and FTIR analyses. Microscopy studies showed that UP resins and their fibreglass composites are porous materials, which increases the area available to physical and chemical degradation. With CIE L*a*b* studies, it was possible to show that the non-stabilised and inexpensive UP products yellowed more strongly during ageing. Yellowing of composites with fibreglass was also more pronounced.

FTIR analyses indicated different chemical stability of commercial UP resin products. The photo-oxidation and cross-linking may continue after the polymer film has cured and may cause brittleness during ageing, which is why UP products to be used for restoration should be carefully tested.

The study also provided useful information for preventive conservation of artefacts made of unsaturated polyester composite materials. Water, humidity, changes in temperature and especially exposure to UV-light cause damage to art objects made of low quality UP composite materials.

“The Cocotte with Two Dogs” has been kept indoors in the Oulu City Museum. Of the installation “The Last Milk Platform”, only some parts (the wooden milk platform and the aluminium churn) are currently kept outdoors. The mermaid, which is made of UP resin composite, has been under conservation and restoration since 2005.

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