Characterization and Stability Issues of Artists' Alkyd Paints

Rebecca Ploeger and Oscar Chiantore

ABSTRACT. Alkyd paints were developed mainly for industrial and household applications and have received minor attention in the art conservation field. The conservation literature is still sparse on the chemistry, characterization, and stability of these materials and even more so on cleaning procedures and conservation recommendations. This does not mean that artists have not used them; rather, they have been (and still are) using them widely. The aims of this paper are to provide a general introduction to these paints and information on how they can be efficiently characterized in works of art, to discuss some of their stability issues, and to give simple recommendations for their conservation. Through a number of analytical techniques the characterization of artists' alkyds and their drying, aging, and degradation processes are explored. On the basis of the information gained and from practical suggestions of conservators, basic guidelines are suggested with the hope that these can be tested and confirmed in the field.

INTRODUCTION

One of the most important classes of binding media of the twentieth century is represented by the alkyd resins. The first alkyds were known as "glyptals" and appeared in the early 1900s. The term alkyd is derived from the polyalcohol and diacid monomers used to make the polyester polymer; the first oil-modified alkyd resins, what we generally refer to as an alkyd today, appeared in the 1920s. However, it was only after the World War II that research and development of alkyds really took off.

Alkyd resins are made by condensation polymerization of polyols, diacids, and siccative oils or free fatty acids, and the final resin is composed of a polyester backbone, which on its own is a brittle, highly branched polymer, and dangling fatty acids, which serve to reduce the amount of cross-linking, creating a more flexible polymer. The polymer is dissolved and carried in a solvent which varies on the basis of the amount of fatty acids present. In the case of alkyd resins for artists' alkyd paints, the resins can be carried in a nonpolar aliphatic hydrocarbon solvent (i.e., mineral spirits).

The main advantage and selling point of artists' alkyds is their rapid drying (18–24 hours, touch dry), regardless of the color (Staples, 1982a), which allows artists to create works swiftly while maintaining some of the aesthetics and working properties of a traditional oil paint (Staples, 1982a, 1982b). The fast drying also makes them ideal for underpainting, impasto, layers, and glazing. The general paint rules that apply to oils also can be applied to alkyds; similar painting grounds can be used, as well as varnishes, brushes, painting knives, and solvents used for cleaning. Artists' alkyds are completely

Rebecca Ploeger and Oscar Chiantore, Department of Chemistry, University of Torino, Via Pietro Giuria 7, 10125 Torino, Italy. Correspondence: Oscar Chiantore, oscar.chiantore@unito .it. Manuscript received 19 November 2010; accepted 24 August 2012.

intermixable with traditional oil paints, and intermediate drying rates can be found by their mixing, so mixed-media paintings can be created without problems (Staples, 1982a). Also, alkyd paints remain soluble in turpentine and white spirits for the duration of a typically working session (6–8 hours); therefore, the initial stiffening can be reversed, and an artist can control the semidry state for long periods of time and make simple modifications. Diluting these paints in solvents can allow an artist to achieve a watercolor painting effect without significant bleeding of the colors into each other (Staples, 1982a).

The type of alkyd resin used in artists' alkyd paints is a longoil alkyd, which is an oil-modified polyester polymer that contains between 56–70 weight percent (wt %) fatty acids. The oil length is the weight percent of fatty acids in the finished resin. Alkyd resins, based on specific applications, are grouped between short oil (35–45 wt %), medium oil (46–55 wt %), long oil (56–70 wt %), and very long oil (above 70 wt %). Changing the oil length and oil type has a great impact on the properties and ultimate applications of the resins; however, many of them are out of the scope for this work, and therefore, only long-oil alkyds will be discussed.

The rapid touch drying of artists' alkyds is due to solvent evaporation (physical drying) and the high molecular weight of the resin, which requires fewer cross-links to form a coherent film. The unsaturation of the fatty acids is, for the most part, unaffected during the manufacturing of the resin, so the chemical drying properties of the alkyd paints are similar to those of traditional oil paints; the cross-linking and film formation takes place by autoxidation, aided by catalysts, such as Co, Zr, and Ca. However, the higher molecular weight of alkyd resins has a disadvantage since alkyds become harder and therefore more brittle at an earlier stage of their aging process than oil colors do (Lack, 1988). Also, long-oil alkyd resins tend to yellow upon aging, especially with linseed oil-based resins that have a high unsaturated fatty acid content. To overcome this, semidrying oils, such as soya, safflower, etc., can be used to manufacture alkyds. The small decrease in drying efficiency is countered by the fact that semidrying oils have a lower tendency to discolor during aging. Figure 1 summarizes alkyd properties obtained with different oils.

As mentioned above, long-oil alkyd film formation occurs by catalyzed autoxidation. During this process, hydroperoxides are formed and degrade into radical species that participate in crosslinking by recombination reactions resulting in ether, peroxy, and carbon-carbon cross-links (Muizebelt et al., 1994; Mallégol et al., 2000). If cross-linking proceeds to an excessive degree, β -scission can occur, forming lower molecular weight degradation products as well as a number of other oxygenated degradation species, such as carboxylic acids, aldehydes, and alcohols. The oxygenated species can change forces within the film and, to a small degree, the film surface interactions with different solvents (Ploeger et al., 2009a). The lower molecular weight species can be lost through evaporation and extraction, contributing

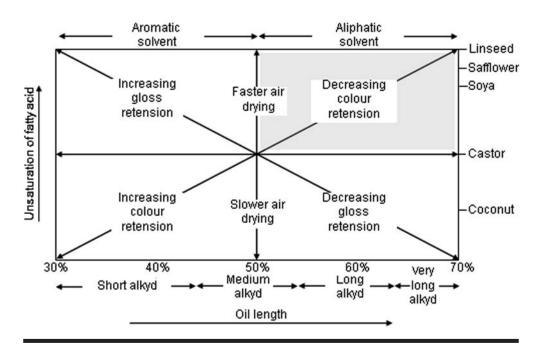


FIGURE 1. Summary of alkyd properties obtained by varying the oil type and oil length of the resin. The shaded top right section represents the area of artists' alkyd resins. Alkyds in the left half are generally for spray and industrial use with lacquer-type drying and curing. Alkyds in the right half are more for brushing applications and have been modified for flowability and better stability in containers. Modified after Patton (1962).

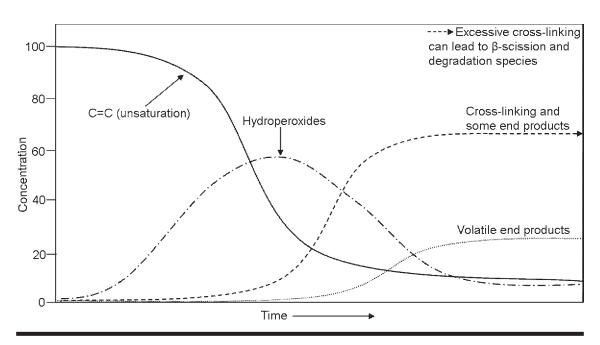


FIGURE 2. Film formation and degradation processes. Modified after van Gorkum and Bouwman (2005).

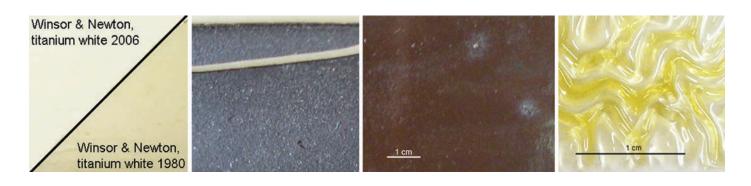


FIGURE 3. Conservation problems encountered with alkyd paints and resins. From left to right: yellowing with age, brittleness (cracking), blooming effect, and wrinkling during drying.

to an increased brittleness of the film since these species could otherwise act as plasticizing agents. Figure 2 describes the general film formation and degradation processes.

Conservation problems encountered with alkyd paints are few but significant in their impact on the stability and appearance of the work of art (Figure 3). The main concerns are (1) pigmentdependent wrinkling and aging of the films, (2) relative humidity and temperature-dependent migration of fatty acids, and possibly other additives, resulting in a "blooming" effect, (3) significant stiffening and cracking of the films with age, and (4) rapid yellowing and darkening.

This paper addresses some of the characterization and stability issues encountered with artists' alkyd paints, which can be comparable to industrial unmodified alkyd formulations. Some practical conservation issues and solutions will be discussed, and basic storage guidelines will be proposed. The results from the artists' alkyd paints are based on studies of Winsor & Newton Artists' Alkyd Colours (discontinued), London Alkyd Colours (discontinued), Griffin Alkyds (currently available) and reference materials, Ferrario Alkyd (Colore Alchidico), Da Vinci Paint Co. Leonardo Oil with Alkyd (formulation from 2006), Paints Dry Quick (PDQ) Quick Dry Oil (discontinued), and various alkyd media.

DISCUSSION

CHARACTERIZATION

In order to identify the presence of an alkyd resin, a number of analytical techniques can be used. One of the most common is Fourier transform infrared spectroscopy attenuated total reflectance (FTIR-ATR). A long-oil alkyd resin can be identified by its characteristic FTIR-ATR spectrum, containing a broad hydroxyl group peak at 3440cm⁻¹ (broad rounded peak), (C-H)CH₂ asymmetric and symmetric stretching and bending peaks at 2,925, 2,855, and 1,465 cm⁻¹ respectively, and a strong C=O stretching peak around 1,726 cm⁻¹ and C-O and fingerprint peaks at 1,256 cm⁻¹ (strong, likely due to esters) and 1,120 cm⁻¹.

The aromatic rings in the polyester backbone can be identified by the sharp doublet aromatic stretching peaks at 1,600 and 1,580 cm⁻¹, the strong unsaturated ring in-plane deformation peak at 1,071 cm⁻¹, and the out-of-plane deformation peaks at 741 and 705 cm⁻¹. Pigments, extenders or fillers, and other additives can complicate spectral interpretation and must be taken into consideration. A typical long-alkyd resin FTIR-ATR spectrum can be found in Figure 4 (for experimental details, see Ploeger, 2008).

In order to disclose the monomers used to manufacture an alkyd resin, pyrolysis–gas chromatography–mass spectrometry (Py-GC-MS) or thermally assisted hydrolysis and methylation (THM)-GC-MS can be applied. Alkyd resins undergo thermal fragmentation, and the resulting monomers, such as phthalic acid (diacid), pentaerythritol (polyol), and individual fatty acids, can be identified. To observe all the components, it is recommended to derivatize the sample since some of the more polar molecules may not elute during the analysis (Ploeger, 2008). Using Py-GC-MS, it was found that the resins containing isophthalic acid (*m*-phthalic acid) as the major diacid in the polyester backbone, such as in the Ferrario, Da Vinci Paint Co., and several PDQ paints, could be confused with other binders since they did not

produce a characteristic peak as does phthalic acid (o-phthalic acid, which elutes as phthalic anhydride), the principal diacid in the Winsor & Newton paints. Online derivatization using tetramethylammonium hydroxide (TMAH) allowed detection and identification of isophthalic acid, as well as all the fatty acids, the polyol (pentaerythritol), and glycerol (if present). The THM-GC-MS pyrograms for these two types of typical long-oil alkyd paint formulations can be found in Figure 4 (for experimental details, see Ploeger, 2008). Additives can also be detected; for example, Figure 4 shows that the Ferrario Alkyd paint contains a phthalate-based plasticizer (peak 10) and that the Winsor & Newton Griffin titanium white contains a hydrogenated castor oil (peaks 11 and 12), likely as a stabilizer or a rheology modifier. A small amount of wax was also found in the Da Vinci Paint Co. paints, and it has been shown that the Winsor & Newton Griffin paints also contain a wax, as well as possibly a small amount of plasticizer, which could be identified only after extraction experiments (Fuesers, 2006).

STABILITY

The most obvious stability issue concerning alkyds paints is their relatively rapid stiffening during aging. This is due to the resin having a high molecular weight and a bulky polymer backbone, so fewer cross-links are required before a coherent film is formed. As chemical drying and film formation progresses, the flexibility of the film decreases. If oxidation reactions continue after film formation has finished, it can lead to the deterioration of the film and its properties. These processes can be excessive cross-linking, chain scission, and the loss of volatile products

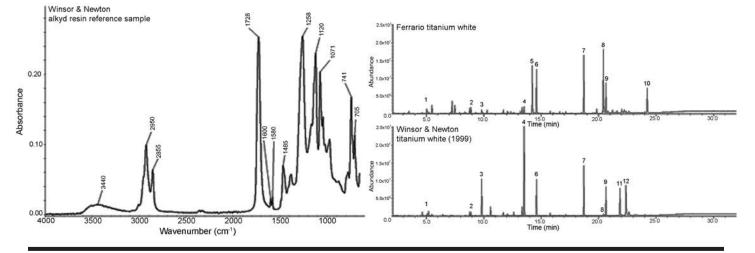


FIGURE 4. (left) The FTIR-ATR spectrum of an alkyd resin reference sample (Winsor & Newton). (top right) THM-GC/MS pyrogram of Ferrario titanium white. (bottom right) THM-GC/MS pyrogram of Winsor & Newton (Griffin) titanium white (cast in 1999). The numbered peaks (all derivatized) refer to (1) a group of glycerol fragments (around retention time $T_r = 5 \text{ min}$), (2) fully methylated pentaerythritol ($T_r = 8.9 \text{ min}$), (3) tri-methylated pentaerythritol ($T_r = 9.8 \text{ min}$), (4) phthalic acid ($T_r = 13.5 \text{ min}$), (5) isophthalic acid ($T_r = 14.2 \text{ min}$), (6) azelaic acid ($T_r = 14.6 \text{ min}$), (7) palmitic acid ($T_r = 18.7 \text{ min}$), (8) oleic acid ($T_r = 20.4 \text{ min}$), (9) stearic acid ($T_r = 20.7 \text{ min}$), (10) phthalate plasticizer ($T_r = 24.3 \text{ min}$), (11) 12-methoxystearic acid ($T_r = 21.9 \text{ min}$), and (12) 12-hydroxystearic acid ($T_r = 22.4 \text{ min}$). Benzoic acid is also present at a low amount around $T_r = 8.4 \text{ min}$.

(Saunders, 1973). These problems can be compared to those of traditional oil paints, which indicate that it is the fatty acid portion of the alkyd that gives rise to many of these issues. If cross-linking continues to an excessive degree, the stiffening of the polymer chains will start to hinder further cross-linking reactions, and the competitive degradation of β -scission will become prevalent. The β -scission results in the formation of a low molecular weight species and radical species. The low molecular weight molecules can remain in the film or be lost by volatilization (Lazzari and Chiantore, 1999) or through solvent treatments. Alkyds are also susceptible to hydrolysis at the fatty acid ester linkages, but not with the same effects or rate as oil paints (O'Neill, 1970). Unfortunately, there is limited information in the literature about the hydrolysis of alkyd paints. The identification (Sonoda, 1998; Schilling et al., 2004; Fuesers, 2006; Ploeger et al., 2008) and extraction (Fuesers, 2006) of azelaic acid in aged films (even after limited aging) indicates that there is some degree of hydrolysis. Free azelaic acid is formed by two separate reaction steps: (1) the oxidation at the C9 position of the unsaturated fatty acid and (2) the hydrolysis of the ester bond (Erhardt et al., 2005).

These processes and their consequences were examined by a number of techniques. The first was an optical contact angle technique that examines the contact angle of a small drop of liquid on the surface of a material (for experimental details, see Ploeger et al., 2009a). The results from these experiments provide information on the polarity and free surface energy of the surface. As an example, if a surface is very hydrophobic (nonpolar), water will bead and not wet the surface; however, if the surface is very hydrophilic (with polar groups), it will spread and wet the surface. In the case of alkyds, the formation of oxidative species during accelerated photoaging was monitored with the assumption that an increase in oxygenated species would increase the polarity of the surface and subsequently change the contact angle of the solvents on the surfaces of the paint films. The results provided insight into the rates of oxygen uptake and oxidative degradation along the film surfaces. This can also be confirmed by following the broadening of the carbonyl peak using FTIR-ATR. The raw alkyd resin showed a linear decrease in the distilled water contact angle, indicating an increase in polarity, after approximately 500 hours of accelerated aging, whereas the alkyd paints showed a plateau between 500 and 1500 hours before starting to decrease. It is assumed that the plateau is an induction period caused by UV stabilizers present in the paint formulations.

In the first 150 hours of accelerated aging, the rapid changes in contact angles are attributed to oxygen uptake into the films. The concentration of hydroperoxy groups involved in autoxidation increases, peaking between 25 and 100 hours depending on formulations, since high amounts of atmospheric oxygen can be absorbed into oil paint films in the first days of drying. After this point, the hydroperoxy groups are consumed as cross-linking and film formation progresses. This phenomenon can be observed with differential scanning calorimetry (DSC), which measures the heat transfer in and out of an entire sample (surface and bulk) over a temperature range (for experimental details, see Ploeger et al., 2009b). For alkyds, the hydroperoxy group decomposition and subsequent recombination reactions are highly exothermic, and this results in an exothermic peak between 100° and 200°C in the DSC pattern. The higher the concentration of hydroperoxy groups is, the greater the peak area is. After approximately 1,000 hours of aging, there is a relatively constant low hydroperoxy group concentration. This is the result of the end of cross-linking, and the small amount of hydroperoxy groups left represents the steady concentration of these groups during the degradation processes. The results for the accelerated photoaging of the Winsor & Newton raw alkyd resin reference are shown in Figure 5. It can be seen that with aging, the surface contact angle decreases, oxygenated species along the surface are formed (FTIR-ATR carbonyl

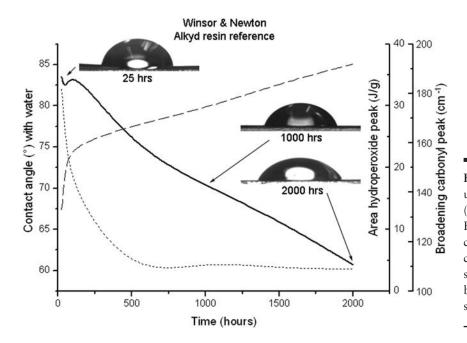


FIGURE 5. Rates of hydroperoxide consumption using DSC (dotted line), decrease in contact angle (solid line), and carbonyl peak broadening using FTIR-ATR (dashed line). The hydroperoxide concentration represents cross-linking, the decrease in contact angle indicates an increase in polarity of the surface of the film, and the broadening of the carbonyl peak indicates the formation of oxygenated species.

peak broadening), and the majority of the bulk cross-linking occurs in about 500 hours (DSC hydroperoxide peak area).

The films become stiff upon aging, and some can be extremely fragile. An increase in film density was observed with thermogravimetric analysis by a small shift in the thermal stability curves to higher temperatures (for experimental details, see Ploeger et al., 2009b). Thermogravimetric analysis provides information about the thermal stability and degradation of a material over a temperature range. A shift in the initial decomposition of the curves represents a difference, in this case an increase, in the thermal stability of the films with age. The stiffening was also observed by changes in the glass transition temperature T_{a} using DSC. Artists' alkyd paints that were naturally aged 1, 8, and 27 years (at the time of analysis) showed an increase in T_{α} with age. The samples cast in 1980 had T_{p} at or above room temperature, which means that with age these films can be brittle at ambient temperatures. The differences in T_{p} of the aged alkyds also suggest a long-term pigment dependence on the aging behaviors of these paints; for example, the Winsor & Newton Artists' Alkyd burnt umber paint from 1980 had a T_{g} of 41°C, whereas the titanium white temperature was around 29°C. The burnt umber pigment likely contains manganese, which will enhance the drying of the alkyd. These phenomena can be correlated back to the excessive cross-linking and subsequent degradation reactions and their consequences. Despite the uptake of oxygen the mass of the organic component of oil paints diminishes during aging, through the loss of volatile scission products (O'Neill, 1970; Lazzari and Chiantore, 1999). The formation of low molecular weight molecules in alkyd media has been demonstrated by O'Neill (1970) and Muizebelt et al. (1994).

PRACTICAL EXAMPLES

To help fill the gap in the literature concerning the cleaning of alkyd paintings, two practical examples will be presented. The first is the painting Spatial Concept by Lucio Fontana created in 1953. This painting has a dark blue glossy alkyd background with holes and glass fragments glued on with a polyvinyl acetate adhesive on a prepared canvas. The alkyd paint, likely an outdoor alkyd house paint, was extremely brittle, and if the canvas support were stressed, the paint layer would crack easily. It was also attracting dust and particulate matter to the surface electrostatically. During the testing of cleaning solvents, it was noted that the painting was extremely sensitive to many traditional nonpolar hydrocarbon and polar solvents used for cleaning; an immediate blooming effect was observed, with an opaque mark left after drying. The blooming effect could be due to solvent penetration and some swelling and/or leaching of additives or low molecular weight molecules from the film. For many solvents, maximum swelling in alkyds is reached within 2 minutes, except for water, which shows a slower swelling rate (Fuesers and Zumbühl, 2008). Cleaning was performed first with a preliminary dry cleaning using Wishab powder removed by a conservation vacuum cleaner, followed by a wet cleaning with a chelating solution (1% sodium citrate aqueous solution) buffered to pH 6.5 to prevent any interference with paint layer. The solution was placed on the paint surface and immediately removed with a surgical micropump without any mechanical action. Finally, the surface was rinsed with deionized water using the same surgical micropump. Retouching was extremely difficult and not very successful since the refractive index and gloss of the alkyd was different from the retouching paints. This is a problem that is also encountered with other glossy alkyd works.

The second example is a painting by Frank Stella, created in 1968, and listed as alkyd on canvas. The paint was applied directly to the canvas and has a matt surface, likely an interior alkyd house paint; the painting consists of two side-by-side sequences of colored bands that spiral to their centers. The alkyd paint in this case did not show any fragility issues since the paint was applied in a thin layer and was also partially absorbed into the unprepared canvas, but it presented dirt, marks, and residue of other materials. After a preliminary dry cleaning with Wishab sponges, the paint was wetted by the above-mentioned chelating solution, which did not cause any blooming effect. On the other hand, other solvents tested produced a lowering of the tone of the colors. The painting was successfully cleaned using a 1% sodium citrate aqueous solution, buffered to pH 6–7, and rinsed with distilled water, using Saugwunder suction block sponges.

CONCLUSIONS AND CONSERVATION RECOMMENDATIONS

The aged alkyd films, whether naturally or artificially aged, were brittle and difficult to handle. This is due to the high molecular weight of the alkyd polymer and faster stiffening from the excessive cross-linking and subsequent degradation reactions. As observed in the practical examples, the mechanical properties and damage can vary on the basis of the type of alkyd, the preparation of the support, and painting technique. Considering general conservation requirements, it is best to handle all alkyd paintings with care, especially those with flexible supports, since they can become very brittle over time. The majority of the problems observed come from the fatty acid component of the resin, so to limit these problems, alkyd paintings should also be stored in conditions similar to those used for oil paintings.

Another point to keep in mind is the potential pigment dependence on the long-term drying and aging behavior of these paints. This point warrants further study. Artists' alkyd paints are designed to be dry to the touch in 24 hours, regardless of the color, but it is clear from the results that it can take much longer for complete chemical drying (autoxidation) and that there are significant differences in T_a of the paints after years of drying.

When identifying and characterizing an alkyd paint using a Py-GC-MS technique, it is important to derivatize the sample since more polar molecular fragments may not elute from the column in all experimental conditions. Derivatization also allows identification of the specific monomers used in the alkyd polymer and could also aid in identifying the brand or type of alkyd paint. For artists' alkyds, Winsor & Newton used resins containing phthalic acid, whereas the Ferrario and the Da Vinci Paint Co. used resins containing isophthalic acid. The PDQ paints contained both types of resins. However, it should always be assumed that the exact formulations of the paints are unknown, because there is a possibility of polymer or additive variations over the years, as well as a change in raw material distributors.

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