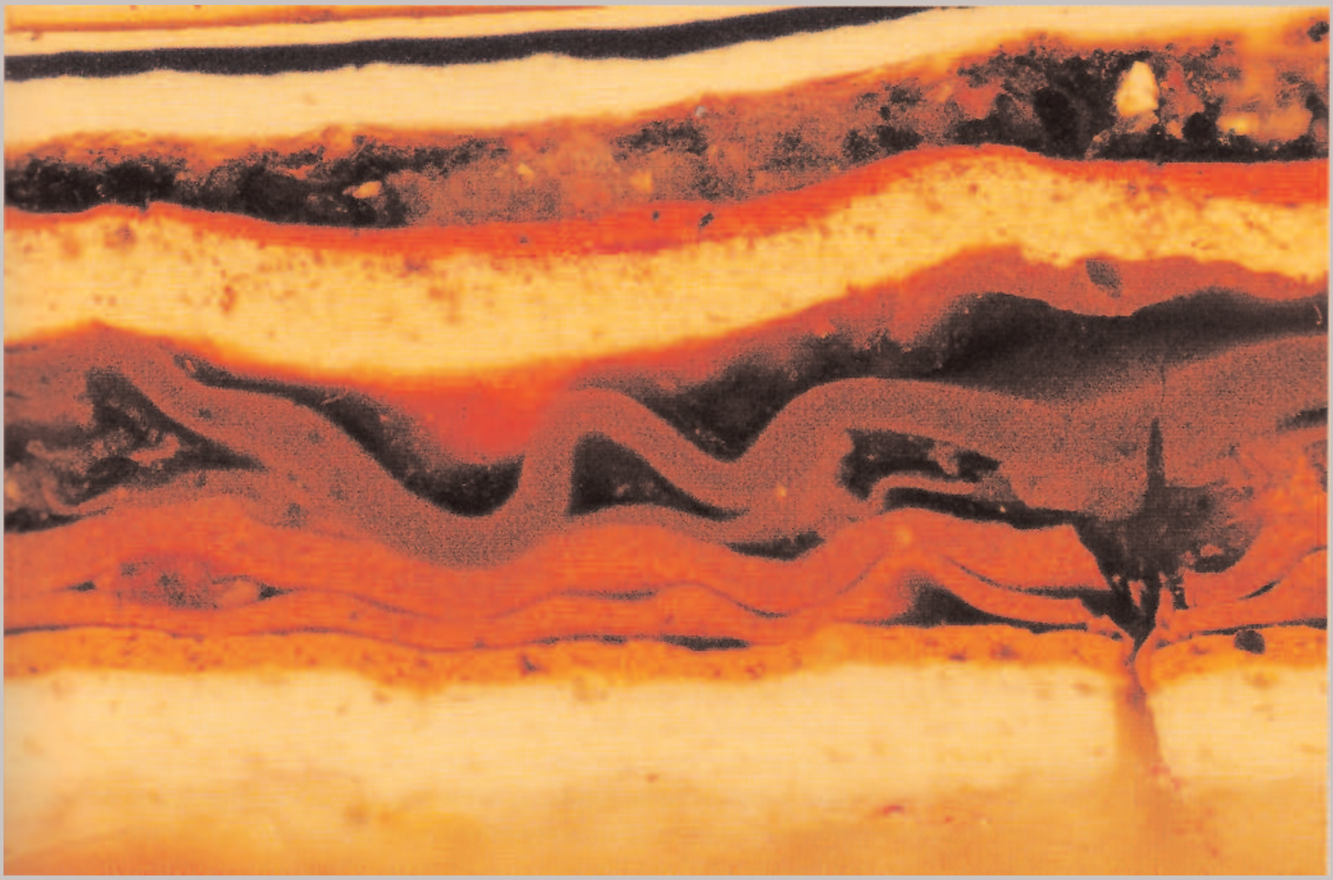
PART SIX

Scientific Research



Structural Response of Painted Wood Surfaces to Changes in Ambient Relative Humidity

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Fluctuations in ambient relative humidity (RH) produce changes in the materials that make up painted wood objects, altering their dimensions and affecting their mechanical properties. The use of wood as a substrate for paint materials presents a particular problem. In the direction parallel to the grain of a wood substrate, applied paint materials are considered to be nearly fully restrained because wood's longitudinal dimension remains essentially unchanged by fluctuations in relative humidity. In the direction across the grain, however, moisture-related movement of an unrestrained wood substrate may completely override the less responsive paint layers. In this situation, stresses induced in the ground and paint layers due to changes in RH are completely opposite to the stresses parallel to the grain.

To quantify the effects of RH fluctuations on painted wooden objects, tests were conducted to determine the individual swelling responses of materials such as wood, glue, gesso, and oil paints to a range of relative humidities. By relating the differing swelling rates of response for these materials at various levels of RH, it becomes possible to determine the RH fluctuations a painted object might endure without experiencing irreversible deformation or actual failure (cracking, cleavage, paint loss) in the painted design layer.

Painted wooden objects are composite structures. They may incorporate varying species of wood, hide glues, gesso composed of glue and gypsum (calcium sulfate) or chalk (calcium carbonate), and different types of paints and resin varnishes. Paint media can include wax, egg tempera, oils, and combinations of these. These materials have differing mechanical properties and varying responses to moisture fluctuations. Painted wood surfaces also vary in complexity. The simplest consist of paint applied directly to wood, but more complex examples, such as early Italian panel paintings in tempera, may have a layer of fabric glued to the wood, a gesso ground to produce a smooth painting surface, paint applied above the gesso, and a clear varnish.

For the past four centuries, oil paint, rather than tempera, has been the most commonly used pigmented coating for wood. Therefore, oil paint on a wood substrate will serve as the focus of this chapter. Hide glue and gesso—materials with distinctive mechanical behavior—will also be included, since they are often incorporated into painted wooden objects. Properties of the individual materials will be discussed here first, followed by aspects of the painted wooden object as a composite structure.

Regarding the effect of RH fluctuations on painted wooden objects, there are three basic conditions of concern. The first, a common construction problem, is built-in restraint that prevents joined wood from naturally swelling and shrinking across its grain in response to RH fluctuations. A variation of this occurs when wood acts as its own restraint (uneven moisture penetration can induce strain into both the dry and wet regions of otherwise unrestrained wood). A second condition of concern is restraint placed on the paint (and any other components of the design layer) by the wooden substrate in the direction parallel to the wood grain (the longitudinal direction). A third and equally important condition involves the RH response of the design layer itself in the direction perpendicular to the grain of an unrestrained substrate. Here, differences in the rate of response of the various materials to changes in ambient RH become especially significant. The following examines these three conditions and identifies the worst cases.

The Wood Support

Over the centuries, many species of wood have been used as paint substrates, with each geographic region favoring certain woods for reasons of fashion, ease of use, and availability. For example, painters from northern Germany and Holland preferred oak while those in southern Germany favored such woods as pine, fir, larch, linden, and ash. In Italy, poplar and cypress were used. American woods, such as cottonwood and mahogany, were imported into Europe for use as supports (Doerner 1962).

Wood responds to moisture by swelling and shrinking with increases and decreases in ambient RH. But wood is anisotropic, meaning that moisture-related dimensional changes vary in its three principal axes—longitudinal (parallel to the grain), radial, and tangential (see Hoadley herein). The most pronounced moisture response is in the tangential direction, where wood may swell up to eighty times as much as in its negligibly responsive longitudinal direction. In the radial direction, wood swells about half as much as it does tangentially (*Wood Handbook* 1974).

If restrained during changes in relative humidity, wood can develop high stresses and strains. If the change in environmental moisture is extreme, the wood may be plastically (permanently) deformed, a condition that can lead to cracking. How much change in relative humidity is necessary to bring about plastic deformation and failure in fully restrained wood and paint materials? The following sections outline experimentation and analysis designed to answer this question.

Mechanical Testing of Woods

Recent research into the response of wood to changes in RH examined the cross-grain mechanical properties of tangentially sawn wood samples of several species (Mecklenburg, Tumosa, and McCormick-Goodhart 1995). Using this testing program, the yield point (the amount of strain necessary to produce permanent plastic deformation) and strength (the amount of stress necessary to cause breakage) of each wood sample could be established. Figure 1a, b shows the stress-strain plots of different samples of cottonwood and white oak, tested at a range of relative humidities. The samples were all incrementally loaded across the grain, and tests were conducted allowing 30 seconds of stress relaxation at each loading point. In this way, time-dependent variations in behavior were greatly reduced. Disparities in the starting points of the tests reflect changes in cross-grain

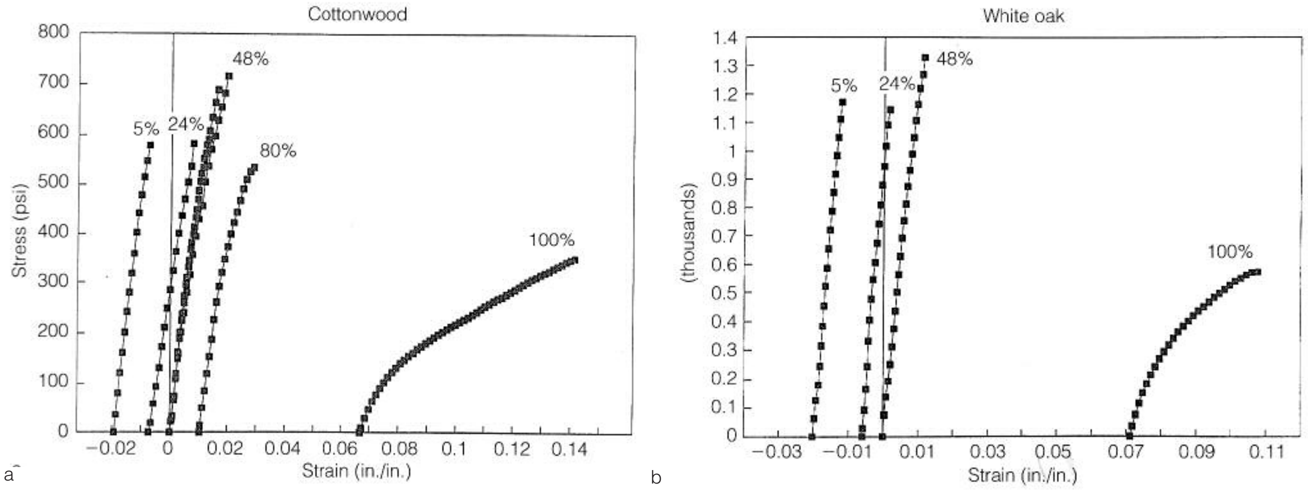


Figure 1a, b
The stress-strain curves for cottonwood (a) and white oak (b), tested at various relative humidities. The space between each plotted test relates to stress-free dimensional change in the sample when the ambient RH is raised or lowered.

dimension, due solely to RH-dependent differences in the moisture content of the wood. The test temperature was approximately 22 °C in all cases. The strengths of the woods are the stresses noted at failure—the end point of each test. Cottonwood, white oak, and American mahogany are strongest at around 50% RH. White oak has the highest strength at around 1340 pounds per square inch (psi).[1]

The amount of strain required to go beyond the elastic (reversible) region to the plastic (irreversible) region can be determined by unloading the specimen at different strain intervals. Testing shows that the yield point of most polymers, including wood, is about 0.004. Figure 2 shows that cottonwood has an initial yield point of 0.004 but, when strain-hardened, it stretches farther.

Environmentally Induced Strains

One can easily recognize that if a hygroscopic material is restrained and desiccated it will experience an increase in stress. What is not so evident is that there is an increase in strain. Too often strain is associated with external deformation when, in fact, no external movement is necessary for strain to occur. Consider a hygroscopic specimen that is allowed to shrink freely from an initial length (L_i) to a final length (L_f) when desiccated from a high relative humidity (RH_h) to a low relative humidity (RH_l). If, under

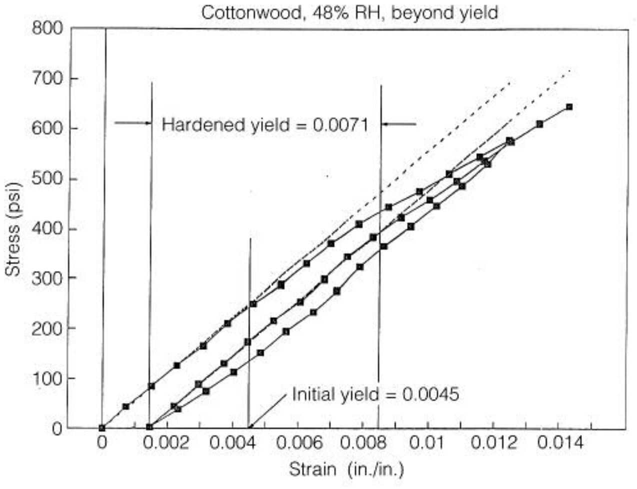


Figure 2
The stress-strain plot of cottonwood that is loaded beyond its yield point of 0.0045 at around 48% RH. After unloading, this specimen exhibits a plastic deformation strain of approximately 0.0014. Reloading indicates an increased yield point of 0.0071.

equilibrium conditions, the specimen is now stretched back to its original length (L_i) it will clearly undergo an increase in stress and strain. It is, however, at the lower relative humidity (RH_l). This new state of stress, strain, and relative humidity is no different from that of a specimen restrained at L_i and RH_h, then desiccated to RH_l without being allowed to shrink (Mecklenburg and Tumosa 1991). The specimens reach identical states following the two different experiments, ending with the same cross-grain dimension and the same relative humidity. The restrained test specimen does not exhibit dimensional change, although it was subjected to an increase in strain. Now it is necessary to determine the amount of change in RH that will cause strains approaching the yield point when a material is fully restrained. If the yield point is not exceeded, then a fully restrained specimen may be subjected without damage to strain induced by variations in RH.

The Swelling Isotherm and the Moisture Coefficient of Expansion

The amount a material swells or shrinks can be expressed as strain (a ratio of the dimensional change of the material to its initial dimension) versus RH. This simply entails holding the temperature constant, measuring the cross-grain dimension (L_{RH}) of a specimen at different RH levels, then establishing an initialized length (L_O), usually the dry length. The strain (ϵ) is calculated as

$$\epsilon = (L_{RH} - L_O)/\ L_O \tag{1}$$

This form of displaying the dimensional response of a material to changes in moisture is useful in relating dimensional properties to mechanical properties of materials. Figure 3a shows the swelling isotherm (at 22 °C) of cottonwood in the tangential direction. This plot shows significant dimensional response to moisture at the extremes of the RH scale and relatively little response to moisture in the region bounded by 30% and 70% RH. The implications are that moisture changes will have the greatest structural effect at extreme low and high RH levels and the least effect in the central RH regions.

It is necessary to determine the allowable RH fluctuations for cottonwood in any ambient RH environment: the RH changes that will not strain the wood beyond its yield point. To do this, one may use the swelling isotherm and simply measure the change in RH that will cause a strain no greater than 0.004 inch (0.01 cm) of dimensional change per inch of initial dimension (the yield point for cottonwood determined in Figure 2). Alternatively, one may determine the rates of dimensional change (*d*) experienced by a material across the entire range of RH from a polynomial fit of the swelling data. These rates, hereafter known as moisture coefficients of expansion (α) are calculated as

$$\alpha = d\epsilon/d\mathrm{RH} \tag{2}$$

The moisture coefficients of expansion for cottonwood are plotted in Figure 3b. Most texts on the swelling of wood report the moisture coefficient as a constant, but Figure 3b demonstrates that this is not the case. This figure also shows that the lowest values of the coefficient of expansion correspond to the flattest portion of the swelling isotherm, namely from 30% to 70% RH.

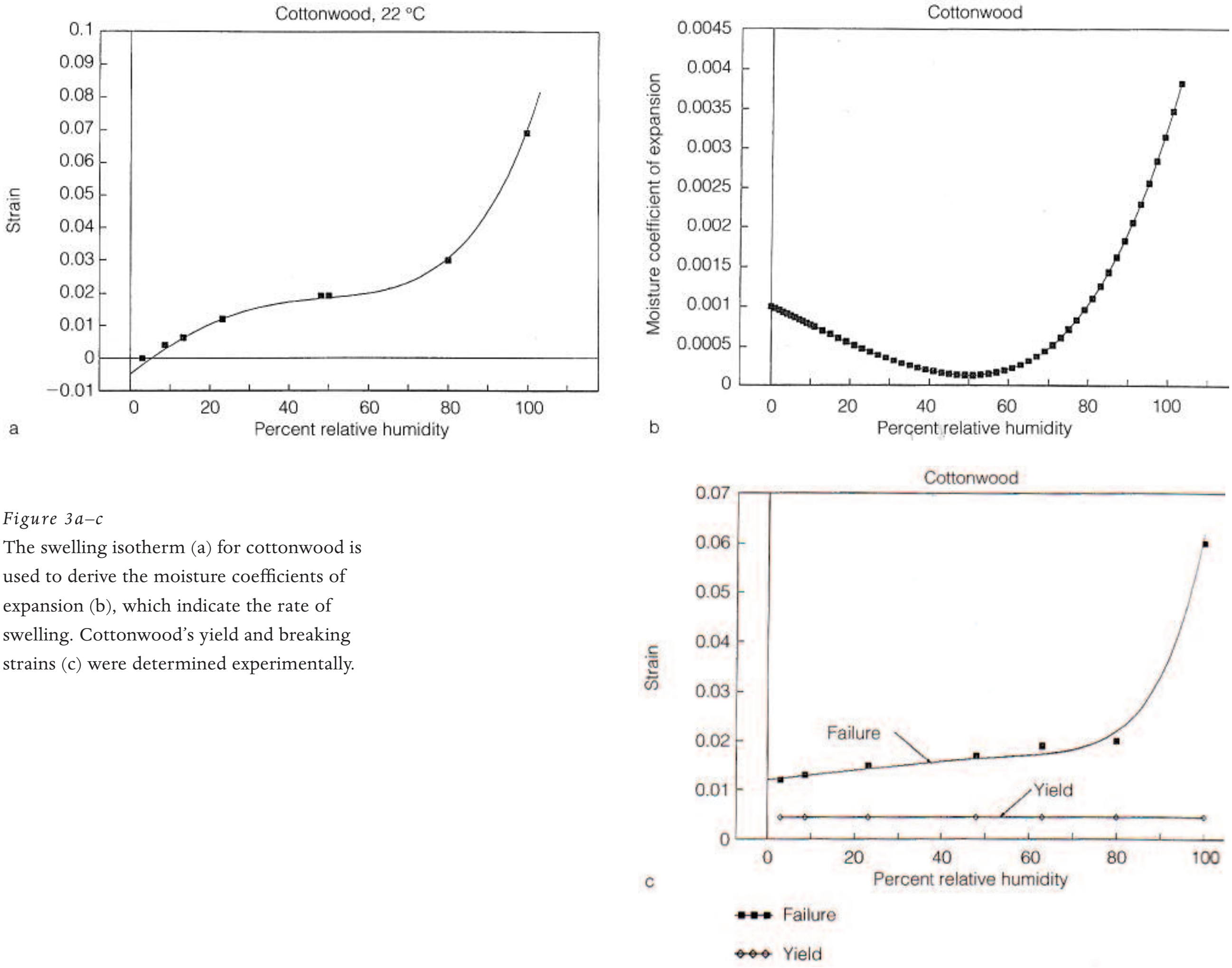


Figure 3a–c
The swelling isotherm (a) for cottonwood is used to derive the moisture coefficients of expansion (b), which indicate the rate of swelling. Cottonwood's yield and breaking strains (c) were determined experimentally.

Using equation 2, the strain change ($\Delta\epsilon$) for any RH change can be calculated as

$$\Delta\epsilon = \int \alpha \, d\mathrm{RH} \qquad (3)$$

where $\alpha = d\epsilon / d\mathrm{RH}$.

To determine the effect of a given strain change on cottonwood, one must know the wood's yield point and the amount of strain necessary to cause it to break. These values are determined experimentally and are shown in Figure 3c. The yield point for new cottonwood is about 0.004 at all RH levels, and its breaking strains increase with increasing RH.

Integrating equation 3 allows one to determine the change in RH that will produce a particular change in strain. One assumes a starting RH of 50% and integrates to another RH level associated with the strain change in question. In the case of cottonwood in the tangential direction, at 50% RH the yielding strain is 0.004, and the breaking strain is 0.017 (Fig. 3c). The associated RH changes required to induce these strains for a sample of cottonwood restrained in the tangential direction (Fig. 4a) are from 50% to 30% RH for yielding in tension, 50% to 67% for yielding in compression, and 50% to 14% for complete failure in tension. No line for complete compression failure is shown in Figure 4a, as compression failures take different forms, from the crushing of the cell walls to buckling

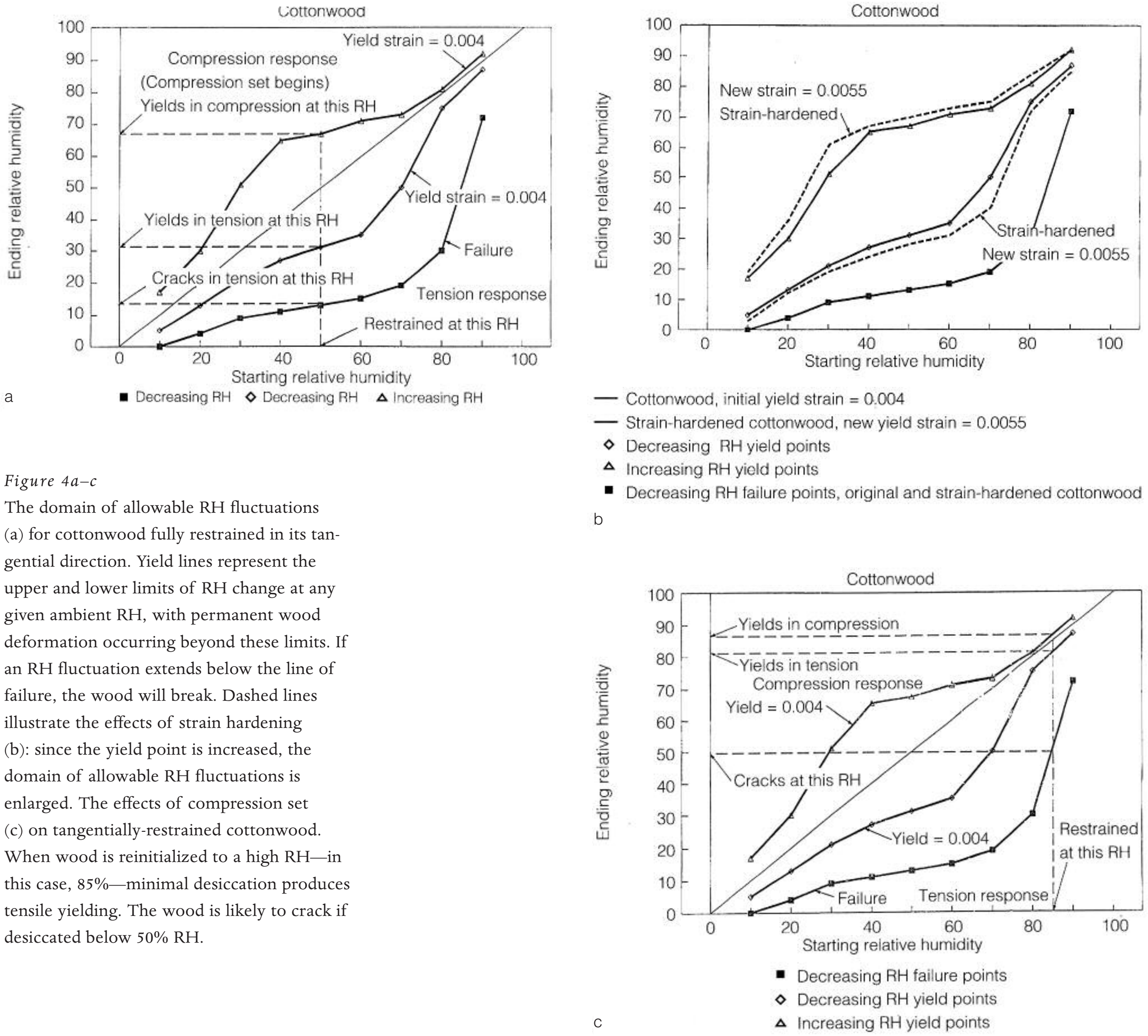


Figure 4a–c
The domain of allowable RH fluctuations (a) for cottonwood fully restrained in its tangential direction. Yield lines represent the upper and lower limits of RH change at any given ambient RH, with permanent wood deformation occurring beyond these limits. If an RH fluctuation extends below the line of failure, the wood will break. Dashed lines illustrate the effects of strain hardening (b): since the yield point is increased, the domain of allowable RH fluctuations is enlarged. The effects of compression set (c) on tangentially-restrained cottonwood. When wood is reinitialized to a high RH—in this case, 85%—minimal desiccation produces tensile yielding. The wood is likely to crack if desiccated below 50% RH.

of the panel itself. Buckling is influenced by the geometry (thickness) and restraint (boundary conditions) of the panel.

Allowable RH Fluctuations

Figure 4a effectively establishes the allowable RH fluctuations cottonwood restrained in the tangential direction may sustain without damage. The tension and compression yield lines set the RH limits (ending RH) from which the ambient RH (starting or equilibrium RH) can deviate. The area between these lines can be viewed as the allowable RH zone. Since the data relate to the wood's tangential direction, this represents the worst-case condition for cottonwood. If the wood were tested in the radial direction, the allowable RH fluctuations would be greater. This is because the yield point is still at least 0.004, but the moisture response of cottonwood in the radial direction is about half that in its tangential direction.

If the wood were wholly unrestrained, Figure 4a would not be pertinent, but in real-world conditions one assumes there is restraint. Restraint may result from basic construction techniques (e.g., wood components are often securely joined with grains mutually perpendicular). Bulk wood also experiences internal restraint when the exterior responds more quickly than the interior to an RH change. Battens and locked cradles on the backs of panels restrain them from freely expanding and contracting. Even under the worst structural circumstances, however, the tests indicate that cottonwood can endure significant RH fluctuations if the ambient RH is centered between 35% and 60%. At higher or lower RH the allowable fluctuations are dramatically reduced. Cottonwood forced beyond the allowable range will experience yielding. If the excursion is severe enough, it is possible that the material cannot return to the central zone without breaking.

If the wood has been strain-hardened, which is probably the case for all old woods, the RH change required to reach the yield point increases. Figure 4b shows the plots for tension and compression when the yield points have increased from 0.004 (solid lines) to 0.0055 (dashed lines). In effect, the allowable elastic or reversible RH fluctuations have been increased. It is important to point out, however, that neither the breaking strength of the material has changed, nor the associated change in RH sufficient to cause cracking.

If a cottonwood sample is restrained at 50% RH and the humidity is increased to 85% RH, the wood will experience plastic deformation in compression, or *compression set*. The wood now has been effectively shortened, and upon desiccation from 85% it begins to experience tension. In effect, the wood has been reinitialized to a restrained condition at 85% RH. Figure 4c shows that, upon desiccation from 85% to about 81% RH, the sample has already attained yield in tension. Upon returning to 50% RH, the sample will most likely crack. This illustration explains why restrained wooden objects subjected to very high humidities or stored outside in equally humid environments often suffer damage when brought into a well-controlled museum environment. The degree of control of the museum environment is thus not the issue. The substantial change from the high humidity to a moderate environment is the cause of failure.

Since wood is most dimensionally responsive in its tangential orientation, tangentially restrained wood is the most vulnerable to RH-induced strain development. Therefore, if one wishes to set a criterion for the allowable RH fluctuations for a restrained panel, one should examine strain development in the tangential direction as the worst case condition. Figure 5 shows the allowable fluctuation plots for white oak in the tangential direction. It is quite similar to the cottonwood plot (Fig. 4a).

The Effects of Wood Variability within a Single Species

Wood samples of the same species can vary considerably in their mechanical properties. It is of interest to know how this variability affects the material's response to fluctuations in the environment. Samples of spruce obtained from two different sources were tested mechanically, with respect to their dimensional response to moisture. The difference in their mechanical behavior was substantial. In Figure 6a, results from tensile tests are plotted as stress-strain curves conducted at 50% RH and 22 °C. The sample A spruce was 4.5 times as stiff and twice as strong as sample B. This difference was more or less consistent over the whole RH range. The yield points for new samples of both woods were 0.004, but the breaking strains were substantially different, as shown in Figure 6b. Even though sample A was stronger, it broke at strains one-third as great as those for sample B, over the entire RH range. When measuring the swelling behavior, the two materials showed little difference, and the moisture coefficients of expansion were quite similar (Fig. 6c). Computing the RH fluctuations that are required to induce yield strains (Fig. 6d) in either compression or tension showed insignificant differences. In addition, these RH fluctuations were greater than those for woods discussed previously in this article. Differences occurred in the RH fluctuation that was required to cause failure in tension. Sample B acted quite

Figure 5
The domains of allowable RH fluctuations for white oak, fully restrained in their tangential directions.

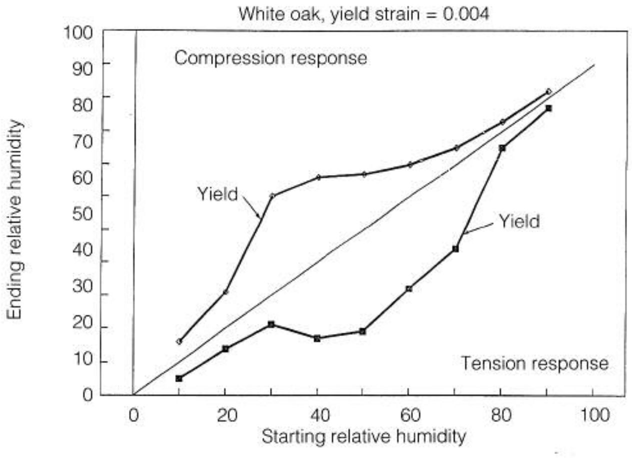
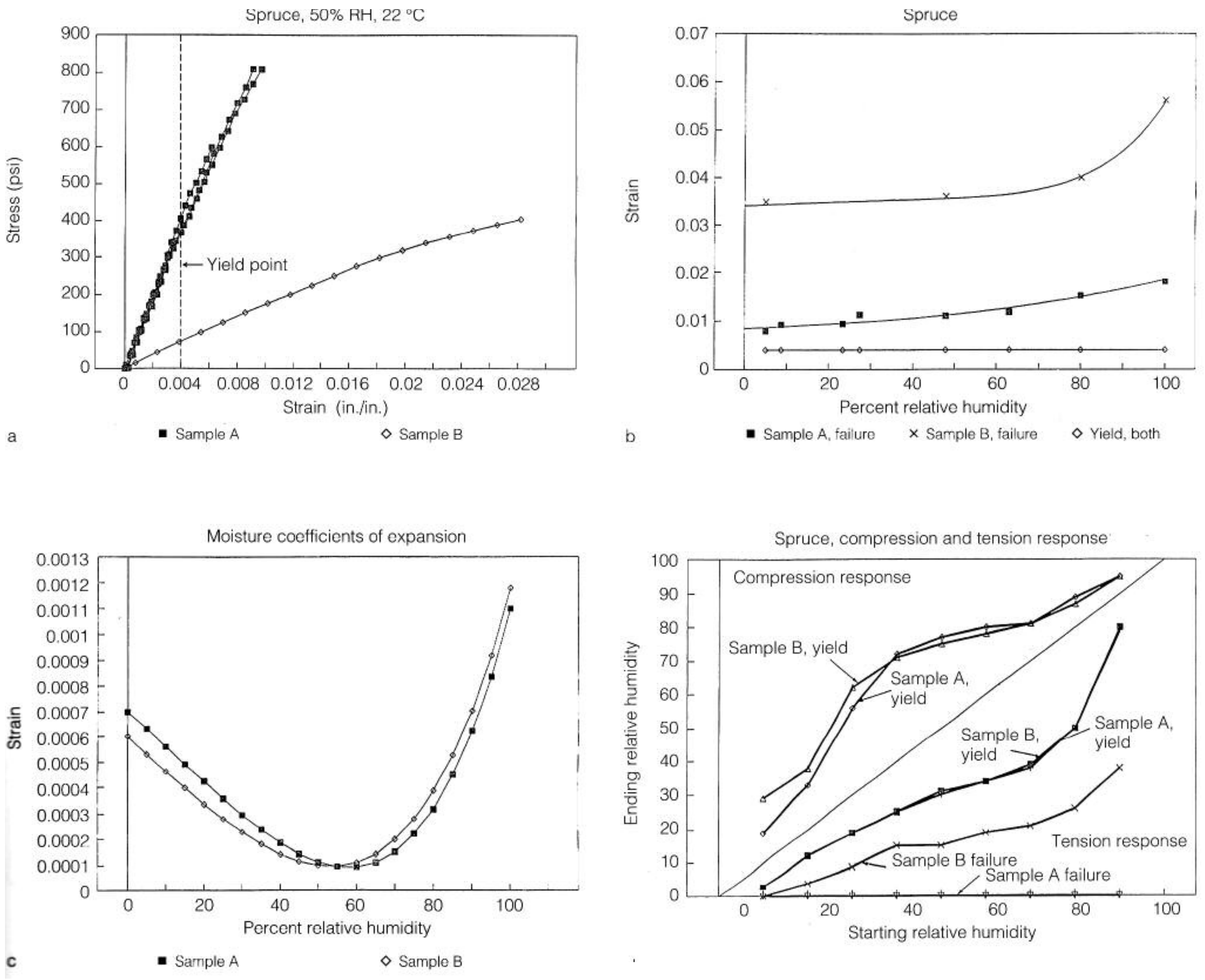


Figure 6a–d
The stress-strain plots (a) and the experimentally measured yield and breaking strains (b) for two samples of tangentially-restrained spruce show substantial variations in mechanical properties. However, the moisture coefficients of expansion (c) and the domains of allowable RH fluctuations (d) are nearly identical.



similarly to the three previous woods, while sample A is difficult to break with any RH change. The interesting aspect of this comparison of two samples of spruce is that mechanical properties, such as stiffness and strength, do not necessarily influence the restrained specimen's yield points (the amount of RH-induced strain required to cause plastic deformation). By contrast, stiffness and strength do influence the restrained specimen's points of failure (the amount of RH-induced strain required to break the sample).

Hide Glue

Hide glue is a material often associated with painted wooden objects, whether used to join the components, to size the surface prior to painting, or as an ingredient in gesso applied to prepare a smooth painting surface. Hide glue is one of the materials most dimensionally responsive to moisture. The swelling isotherm for a particular type of hide glue (rabbit-skin glue) was developed from two separate samples of the same material (Fig. 7a). The newly cast material shrinks freely when initial high RH levels are reduced (Mecklenburg and Tumosa 1991). The total shrinkage can be as much as 6% when desiccating from 90% to 10% RH.

Over short time spans—days to weeks—one can measure a yield point of about 0.004 to 0.005 for rabbit-skin glue (the breaking strains are measured at approximately 3% to 4%—strains of 0.03 to 0.04—in extremely slow tests). For changes in RH that occur within these short periods, it is possible to determine the RH fluctuations that will induce yield strains in restrained hide glue (Fig. 7b). In effect, at 50% RH, the short-term RH fluctuations may range ±11% before yield point is reached. Since hide glue has no covalent cross-links, it is prone to stress relaxation over periods ranging from six to twelve months, depending on the RH level (Mecklenburg and Tumosa 1991). Therefore, over a long period of time any induced stresses may relax. If subjected to excessive RH (over 85%), however, the glue reactivates; as the reactivated glue desiccates with decreases in RH, extremely high stresses develop.

Hide glue response when attached to an unrestrained wood support

Theoretically, rapid extreme changes in RH could cause significant damage to objects sized with glue, due to the resulting high stresses. While such

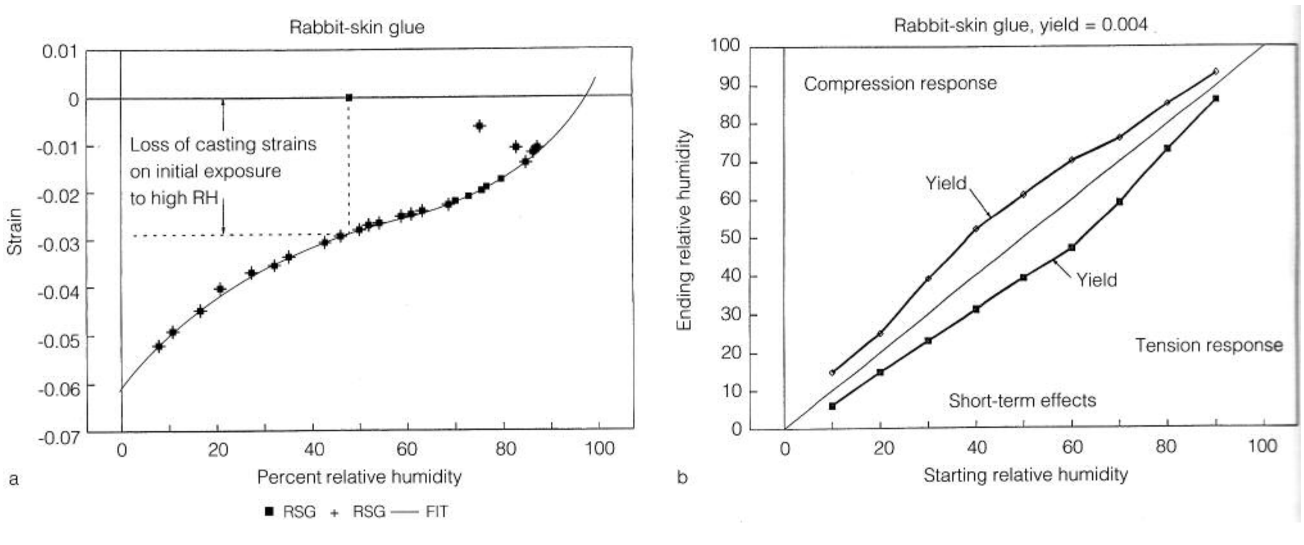


Figure 7a, b
The swelling isotherm for rabbit-skin glue (a) and the domain of allowable RH fluctuations for fully restrained rabbit-skin glue (b) at any given ambient RH.

damage is common in paintings on sized canvas, the mass of the substrate in a glue-sized painted wooden object provides substantial resistance to stresses developing in the relatively thin layer of glue. Since wood has a very small dimensional response to moisture in its parallel-to-grain (longitudinal) direction, wood in this axis essentially serves as a full restraint for all materials attached to it—glues, fabrics, gessoes, and paint.

Across its grain, unrestrained wood will change dimensionally with RH changes. If the moisture coefficients of expansion of all materials attached to a wood substrate were the same as the wood, then RH changes would induce no cross-grain stresses in the attached layers. In reality, the expansion coefficients of the different materials vary considerably, but by comparing them it is possible to explore the effects of RH changes in the cross-grained direction of an unrestrained painted panel. Figure 8a compares the expansion coefficients of cottonwood and hide glue. At approximately 70% the plots intersect: at this RH, both wood and glue swell or shrink at the same rate. In the RH range from 35% to 60%, the glue attempts to swell or shrink at a slightly higher rate than the wood substrate. In this region, glue applied to cottonwood will develop stresses, but only a fraction of those that result from full restraint, since the shrinking or swelling of the wood approximates that of the glue. This is an example of partial rather than full restraint. In effect, the wood's movement provides greater RH tolerances for the hide glue. The strains in the glue can be calculated using the following equation:

$$\Delta\epsilon_G = [(1 - \int\alpha_S d\mathrm{RH}) - (1 - \int\alpha_G d\mathrm{RH})]/(1 - \int\alpha_S d\mathrm{RH}) \qquad (4)$$

where α_S is the coefficient of expansion of the substrate, and α_G is the swelling coefficient of the hide glue.

This equation can be used for any material applied to any substrate in any direction. For example, assume that the coefficient of expansion for the substrate is zero. In that case, Equation 4 simplifies to Equation 3. In the examples to follow, cottonwood will be the substrate since it is one of the most dimensionally responsive woods.

The calculated increase in RH tolerance for hide glue size on a wood substrate is illustrated in Figure 8b. Here, the hide glue is applied to an unrestrained cottonwood support, and the glue strains are plotted

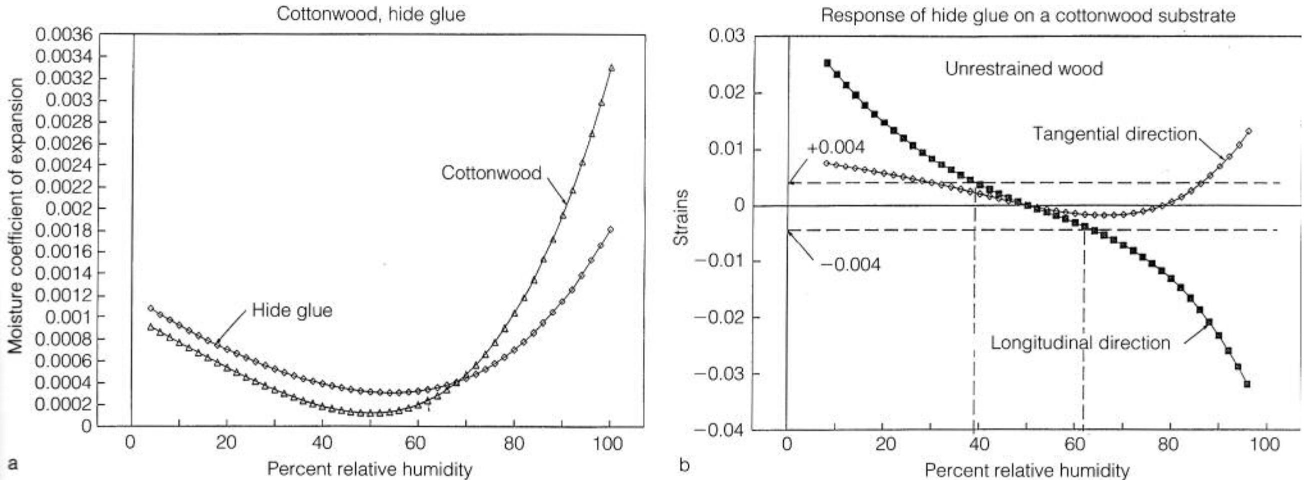


Figure 8a, b

A comparison of the moisture coefficients of expansion (a) for hide glue and cottonwood. Using Equation 4, these values enable one to determine the strain interaction of the glue with the cottonwood substrate (in both tangential and longitudinal directions) over the complete range of RH (b).

against RH in both the tangential and longitudinal directions. The longitudinal direction, in which the glue is essentially fully restrained (the wood's coefficient of expansion in that direction is assumed to be zero), is plotted by integrating Equation 4 from 50% RH going in both increasing and decreasing directions. The strains are as one would expect for fully restrained glue: high in tension (positive values) upon desiccation, and high in compression (negative values) upon increases in humidity. To determine the strains in the glue in the wood's tangential direction, Equation 4 was integrated again, now using cottonwood's tangential coefficients of expansion, shown in Figure 8a. In this direction, the wood substrate and the glue respond similarly to the moisture changes, significantly reducing the strains in the glue layer. With desiccation, the glue strains are in tension but are less than half those in the longitudinal direction. This is because from 50% to 0% RH, the glue coefficient is greater than that for the wood. Increasing the humidity from the 50% RH starting point produces different results. At about 68% RH, the wood coefficient becomes greater than the glue; the swelling of the wood actually overrides that of the glue, and the wood begins pulling the glue layer into tension.

Regarding allowable RH fluctuations, the strains upon glue in wood's fully restrained, longitudinal direction are the most severe, but it is clear that severe desiccation could cause cracking in the glue in both of the wood's directions. In addition, extreme humidification simultaneously subjects the glue to significant tension in the wood's tangential direction and severe compression in its longitudinal direction—both strains potentially leading to failure. Similar diagrams will be used to examine the response of gesso and paint layers attached to wood substrates.

Gesso

Gesso is a mixture of a hide glue and gypsum or ground chalk, used to prepare a smooth, paintable surface on wood. Sometimes other inert materials, such as zinc white and clay, are incorporated. The ratio of inert solids to hide glue has a dramatic influence on the mechanical and dimensional properties of the gesso. This ratio can be expressed in the pigment-volume concentration (PVC). The higher the concentration of inert filler (higher PVC), the weaker, stiffer, and less dimensionally responsive to ambient moisture a gesso becomes, due to the smaller relative amount of glue. Figure 9a plots the dimensional response against RH for two gesso mixtures: PVC = 58.3, and PVC = 81.6. The lower PVC gesso has a maximum change of about 1.5% (strain = 0.015) over the entire RH range. The higher PVC gesso changes only about 0.6% over the same RH range, which is about one-tenth that of pure hide glue (in effect, PVC = 0) which swells as much as 6%. With the increased PVC, gesso will reach yield point and experience failure at dramatically lower strains, and the reasons for this were discussed in a recent study (Michalski 1990). Figure 9b shows the difference in the moisture coefficients of expansion for the two gessos. Both show lowest values (representing the lowest rates of dimensional response) between 50% and 60% RH.

As with all the materials examined, RH affects the mechanical properties of gesso. Tensile testing of one of the gessoes (PVC = 58.3) shows a dramatic loss of strength with increasing RH (Fig. 9c). Here the low RH strains for the gesso tested are actually higher than those determined in the midrange RH tests. The yield strains are about 0.0025, and

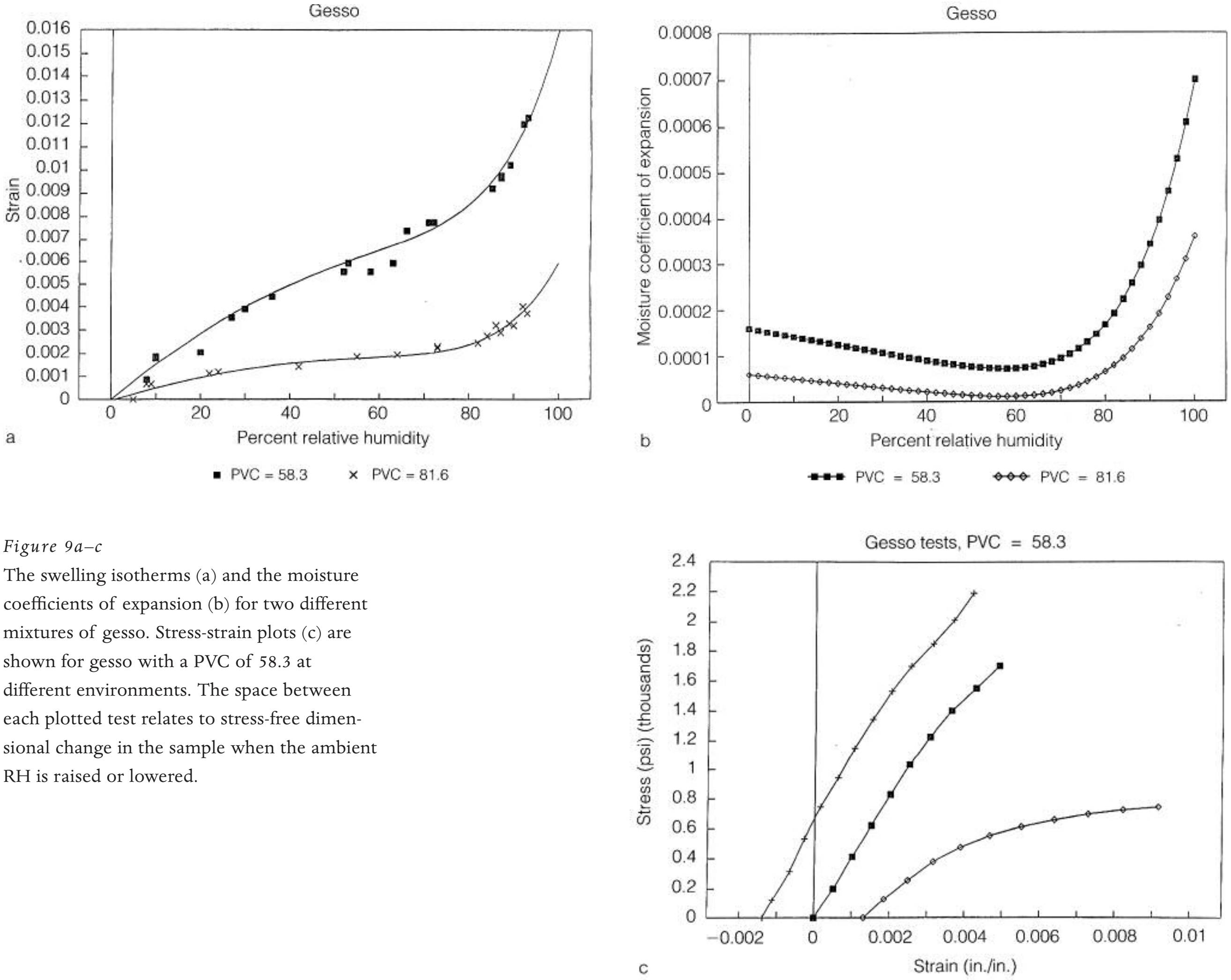


Figure 9a–c

The swelling isotherms (a) and the moisture coefficients of expansion (b) for two different mixtures of gesso. Stress-strain plots (c) are shown for gesso with a PVC of 58.3 at different environments. The space between each plotted test relates to stress-free dimensional change in the sample when the ambient RH is raised or lowered.

the breaking strains again vary with RH but are generally lower than those for other materials associated with painted wood.

In spite of its low yield and breaking strains, gesso can be subjected to greater RH changes than most of the other materials under consideration before reaching its yield points. Figure 10a shows the RH changes required to induce yield and cracking in gesso (PVC = 58.3) fully restrained in the longitudinal orientation of a wood substrate. For example, beginning at an ambient RH of 50%, fully restrained gesso can desiccate to 26% RH before attaining yield in tension. The RH can go as high as 76% before yield is reached in compression. This is a direct result of the gesso's low moisture coefficient of expansion.

Figure 10b compares the swelling coefficients of gesso with those for cottonwood in its tangential direction. Here, the swelling coefficients of the cottonwood and the gesso are not only very low but nearly identical in the 40–60% RH range. This means that an unrestrained cottonwood panel and its applied gesso are swelling and shrinking very little and at almost exactly the same rate in this RH region; there is effectively little structural interaction.

Deviation from the mid-RH zone, however, has quite dramatic effects. Equation 4 was again integrated in order to explore the composite

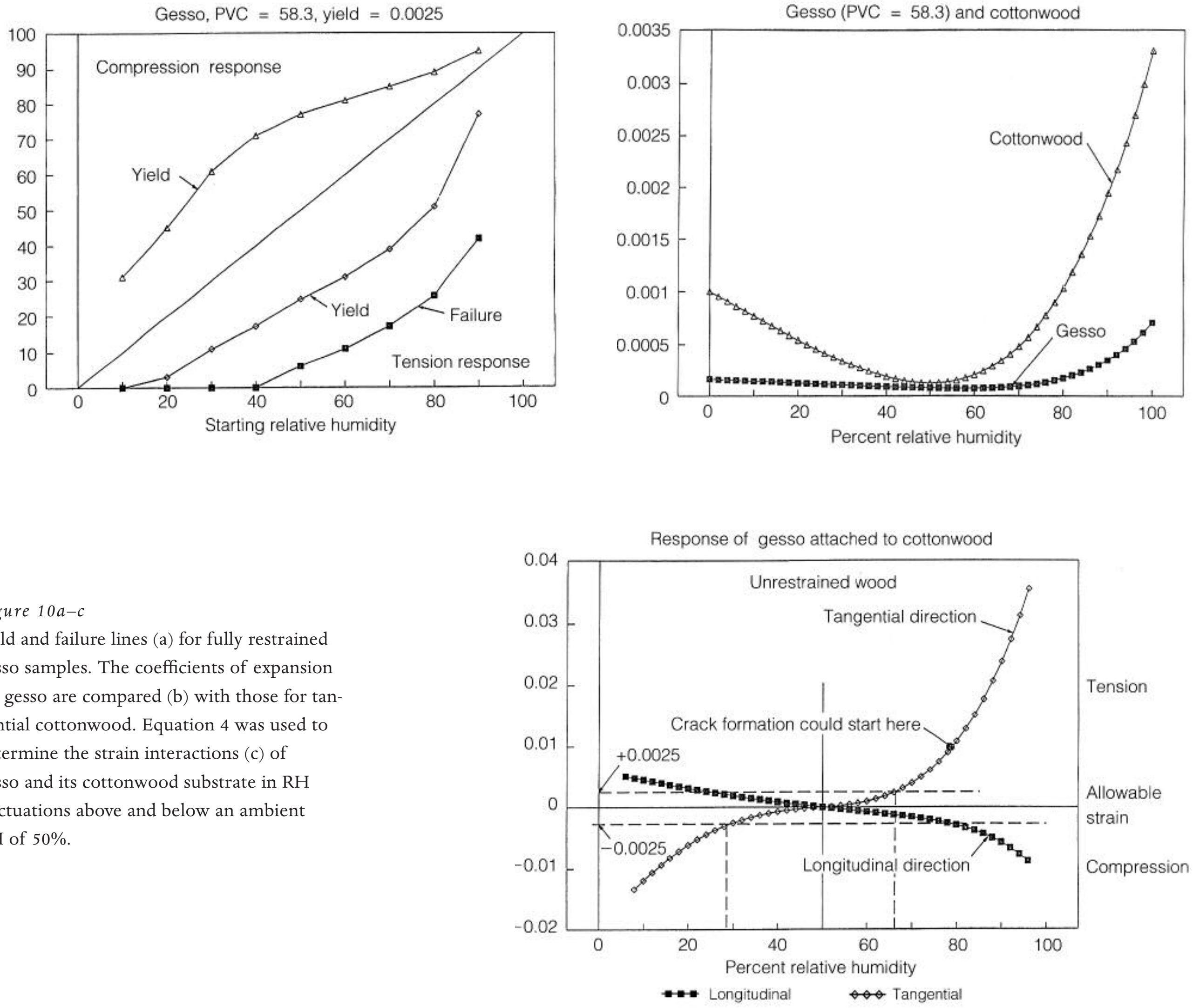


Figure 10a–c
Yield and failure lines (a) for fully restrained gesso samples. The coefficients of expansion for gesso are compared (b) with those for tangential cottonwood. Equation 4 was used to determine the strain interactions (c) of gesso and its cottonwood substrate in RH fluctuations above and below an ambient RH of 50%.

effect when the gesso is bonded to a cottonwood panel, using a starting point of 50% RH. The results of this analysis are illustrated in Figure 10c, where gesso strains associated with both the tangential and longitudinal directions of the wood support are plotted against RH. In the longitudinal direction, full restraint is again assumed, and the RH fluctuations needed to induce yielding are quite large—from 50% to 26% RH in tension upon desiccation and from 50% to 76% RH in compression upon humidification. In the tangential direction, the mismatch of swelling coefficients for the wood and the gesso worsens dramatically as the high and low extremes of the RH range are approached (Fig. 10b). Consequently, Figure 10c demonstrates that desiccation will induce severe compressive strains in the gesso layer (which can cause cleavage and buckling), while raising the RH from 50% to 80% can actually cause the gesso layer to crack. Thus, the range of allowable RH fluctuation is more limited for the tangential direction than for the longitudinal direction. Nonetheless, from a starting point of 50% the RH can range downward to 28% or upward to 66% without causing the gesso to yield in the tangential direction. Even in this worst-case example, the gesso/wood composite can survive significant fluctuations in RH.

The Paint Layers

To study stiffness, strength, and response to relative humidity fluctuations, mechanical tests were carried out on fifteen-year-old oil paints under true equilibrium conditions (several weeks of stress relaxation occurred prior to any subsequent incremental loading). Figure 11a, b shows the tensile test results for two of these paints. Although the yield points for nearly all the paints remained at about 0.004 throughout the tests, the breaking strains varied from one paint to another when tested at the same RH and temperature. For example, at 48% RH and 22 °C, flake white in safflower oil attained a breaking strain of 0.02 (Fig. 11a); titanium dioxide in safflower oil broke at 0.01 (Fig. 11b). When the same paints were tested at different relative humidities, it became apparent that RH plays a considerable role in modifying the mechanical properties. Flake white in safflower oil is a fairly flexible paint that was not seriously affected by the change in RH from 48% to 5% (Fig. 11a). Meanwhile, titanium dioxide in safflower oil lost nearly all its stiffness and strength when the RH was raised from 48% to 80% (Fig. 11b). In general, increasing the RH decreased the strength of a paint but increased its breaking strain, while lowering the RH increased its strength but lowered the breaking strain.

Typically, oil paints are far less dimensionally responsive to moisture than are hide glues or wood. Flake white in safflower oil, for instance, shows a total change in length of only about 0.5% when the RH is raised from 20% to 90%. A plot of the moisture coefficients of expansion (Fig. 12) demonstrates that this is one of the least dimensionally responsive materials presented in this discussion.

Effects of solvents on paint

Leaching out the soluble components of oil paints has a dramatic effect on the mechanical properties. Two fifteen-year-old paints, cadmium yellow in safflower oil and cadmium yellow alkyd artist's paint, were soaked in toluene for a week, then allowed to dry for eight weeks. Figure 13a, b shows results of equilibrium tensile tests before and after toluene treatment for the oil and alkyd paints, respectively. Surprisingly, the yield points of the toluene-treated paints were no different than those for the untreated paints. After treatment, however, each paint experienced a

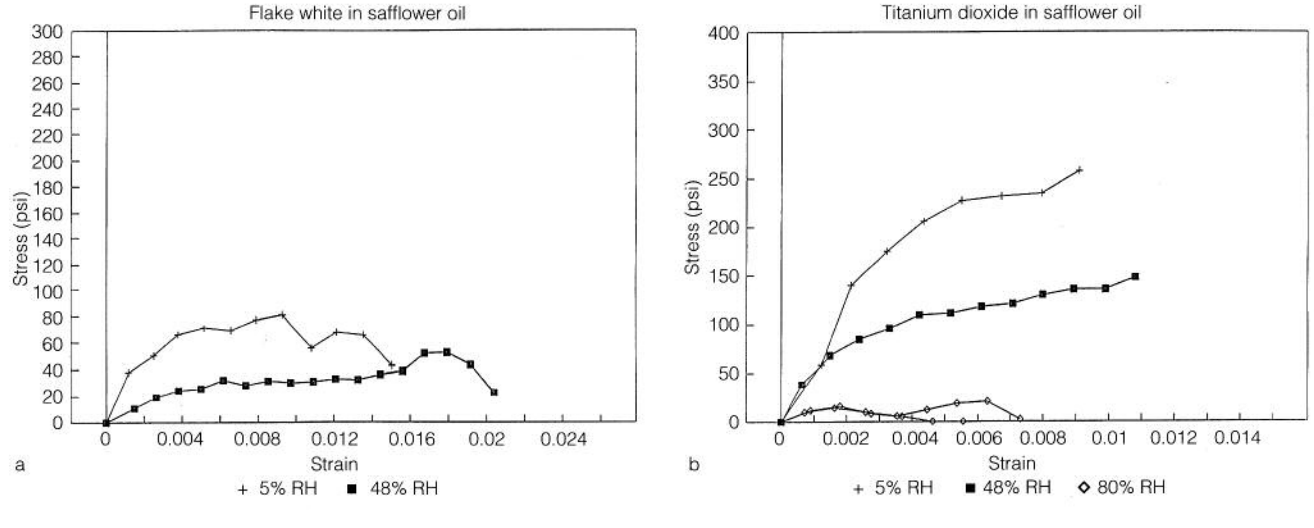


Figure 11a, b

The equilibrium stress-strain plots at different environments for flake white (a) and titanium dioxide (b), both in safflower oil.

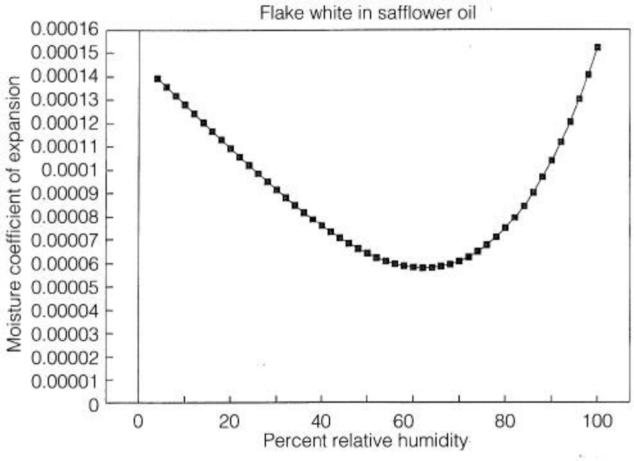


Figure 12
The moisture coefficients of expansion for flake white in safflower oil are the lowest of any included in this study.

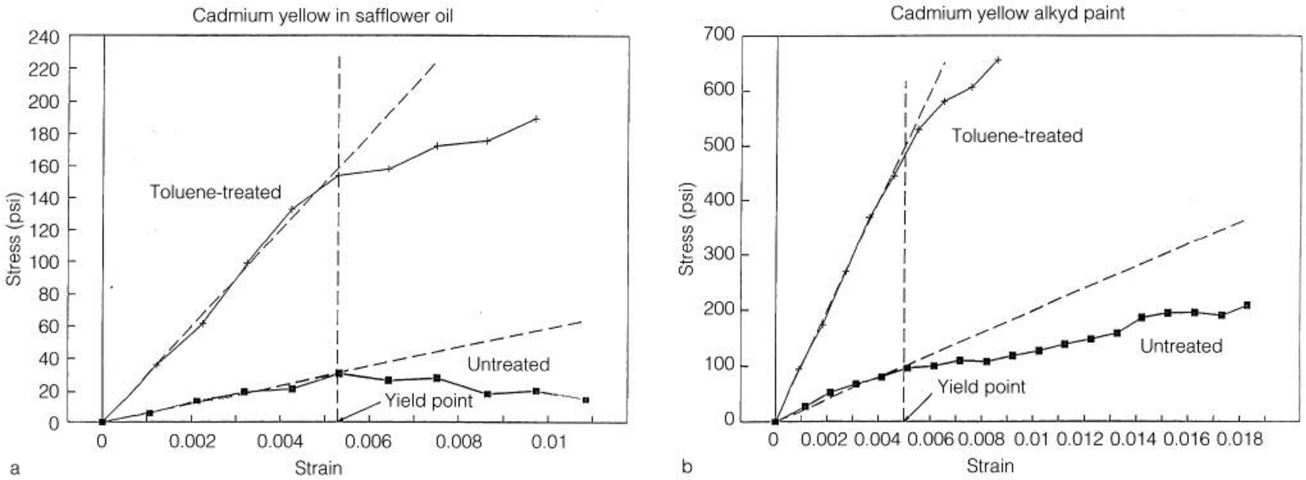


Figure 13a, b
The equilibrium stress-strain curves for cadmium yellow in safflower oil (a) and in alkyd (b), before and after soaking in toluene for one week.

fivefold increase in stiffness and at least a threefold to fourfold increase in strength. Obviously, the soluble components leached out by the toluene had been acting as plasticizers. Solvent leaching simulates one of the possible aging processes of oil paints that results in increased stiffness and strength: the slow evaporation of free fatty acids and other volatile, low molecular weight components (Michalski 1990; see also Erhardt herein).

There is no difference in the swelling characteristics of the treated and untreated paints. The moisture coefficients of expansion in treated oils and alkyds do not change from their untreated values (Fig. 14a, b).

Using the strain-to-yield values and integrating the expansion coefficients for the paints tested, it is possible to establish the allowable RH fluctuations for several restrained paints. Figure 14c shows the acceptable ranges for cadmium yellow in oil (toluene-treated and untreated), cadmium yellow in alkyd (treated and untreated), flake white in oil, and titanium dioxide in oil. Since the toluene treatment altered neither the yield points nor the expansion coefficients of the paints tested, the allowable RH fluctuations are no different for treated and untreated paints. The difference, if any, will be reflected in any changes in strain to break caused by the solvent.

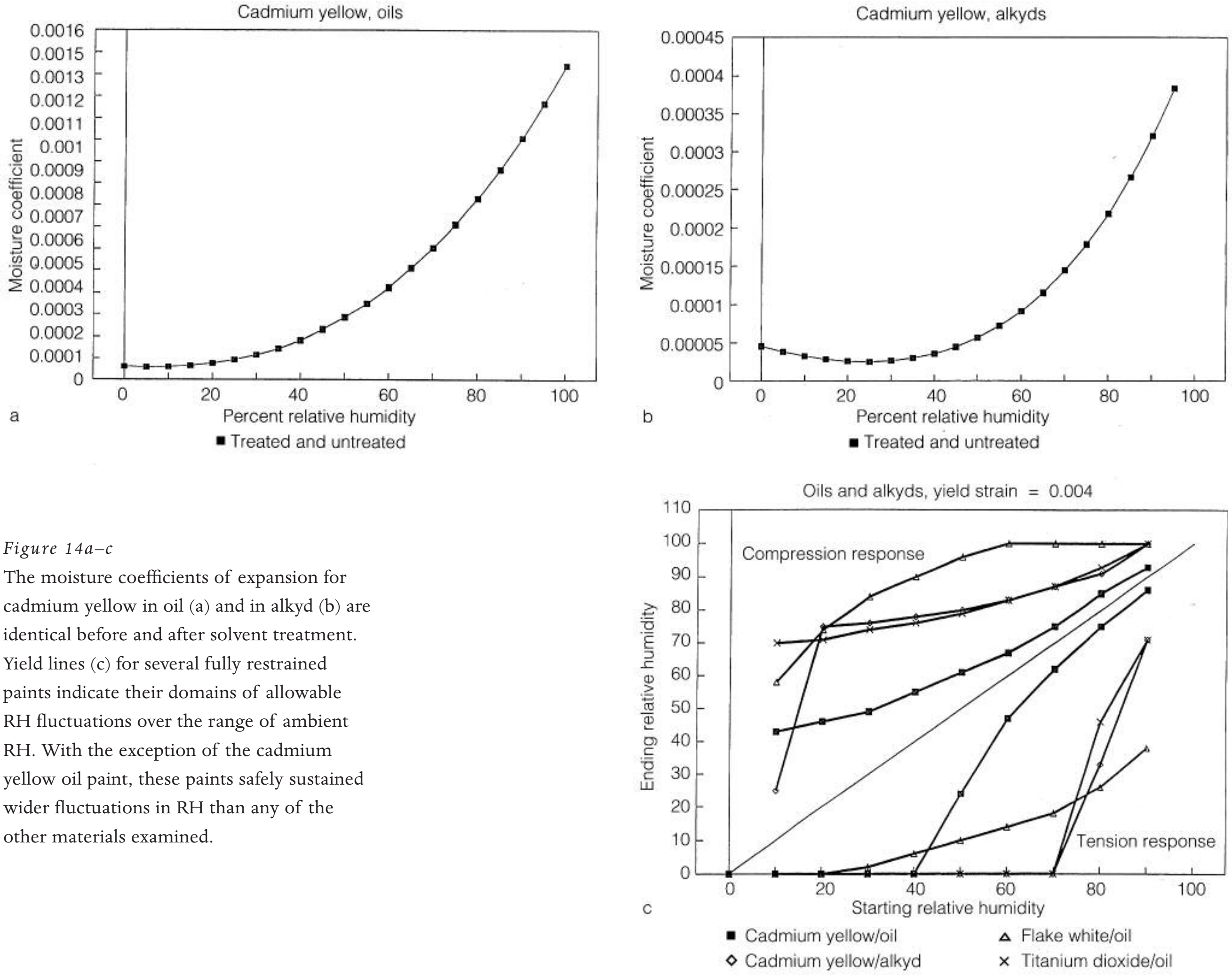


Figure 14a–c
The moisture coefficients of expansion for cadmium yellow in oil (a) and in alkyd (b) are identical before and after solvent treatment. Yield lines (c) for several fully restrained paints indicate their domains of allowable RH fluctuations over the range of ambient RH. With the exception of the cadmium yellow oil paint, these paints safely sustained wider fluctuations in RH than any of the other materials examined.

Response of a composite painted wood surface

For purposes of comparison, the moisture coefficients of expansion for cottonwood, hide glue, gesso, and flake white oil paint are plotted in Figure 15a. The very low coefficients of oil paint and gesso are almost identical, except the coefficients for gesso rise a bit in the range from 70% to 100% RH, while those for the paint stay nearly flat. As with the gesso, the paint—when applied to an unrestrained wood substrate—will experience a serious swelling mismatch outside of the midrange RH. This mismatch can be demonstrated by using equation 4 to calculate the strains expected in a composite of cottonwood, and flake white in oil. Figure 15b shows the flake white strains initialized at 50% RH. In the longitudinal direction, the allowable fluctuations for the paint are quite large. With desiccation from 50% RH, the RH can drop to about 10% before the paint reaches yield point. With humidification above 50%, the RH can rise safely to about 95% RH. In the tangential direction, desiccation to 26% RH will cause compression yielding; beyond that, buckling and cleavage can occur. Increasing the RH to 70% will cause yielding in tension, with possible cracking at extremely high RH levels.

Flaking and the Adhesion of the Design Layers

Normally, the physical separation of the design layer from the support (i.e., peeling, curling, or flaking) manifests after a crack has occurred. There are occasions, however, when the paint layer separates from the wooden support without an associated crack. In art conservation this is usually referred to as *blind cleavage,* while the paint industry calls it *blistering* of the paint. On exterior painted wooden (and metal) surfaces, blistering may result from the solar heating of soft, freshly painted surfaces, from the penetration of excessive amounts of moisture, or from the generation of a gas beneath the paint layer (Hess 1965:102–4; *Wood Handbook* 1987:16–21; Houwink and Salomon 1967:51; Van Laar 1967). In all probability, poor preparation of the substrate also contributes to interlayer or blind cleavage caused by exposure to excessive moisture. Lean (insufficient glue) gesso grounds are particularly susceptible to blind cleavage because of the low cohesive and adhesive strength of the material.

A great number of causes have been proposed for the flaking of a previously cracked paint film. Exterior paints may flake because of exposure to frequent cycles of wetting (by rain) and drying. Too little oil binder may cause poor adhesion of the paint, as may contamination of the prepared surface with dirt, wax, fats, nondrying oils, or grease. Cold temperatures may also cause flaking. As Hess states (1965:242):

> It is widely known that elasticity, adhesion, and shock resistance of most paints and varnishes, even those of very long-oil length, suffer badly during cold weather. The temperature need not necessarily be below freezing point and susceptibility already exists for some coatings at about 50 °F (10 °C). As long as the films are on rigid structures and do not crack the failure does not become permanent and is hardly apparent at all.

Certain woods accept and hold paint better than others. Cedar, cypress, and redwood are excellent woods for painting, while red and white oak, elm, chestnut, and butternut are considered poor choices. In fact, many of the woods historically used as paint substrates for art objects, such as cottonwood, basswood, and yellow-poplar, are not recommended as suitable for exterior painted finishes in the *Wood Handbook* (1987:16–21), one of the standard industrial texts. One of the factors affecting the permanence of a painted surface is moisture-related dimen-

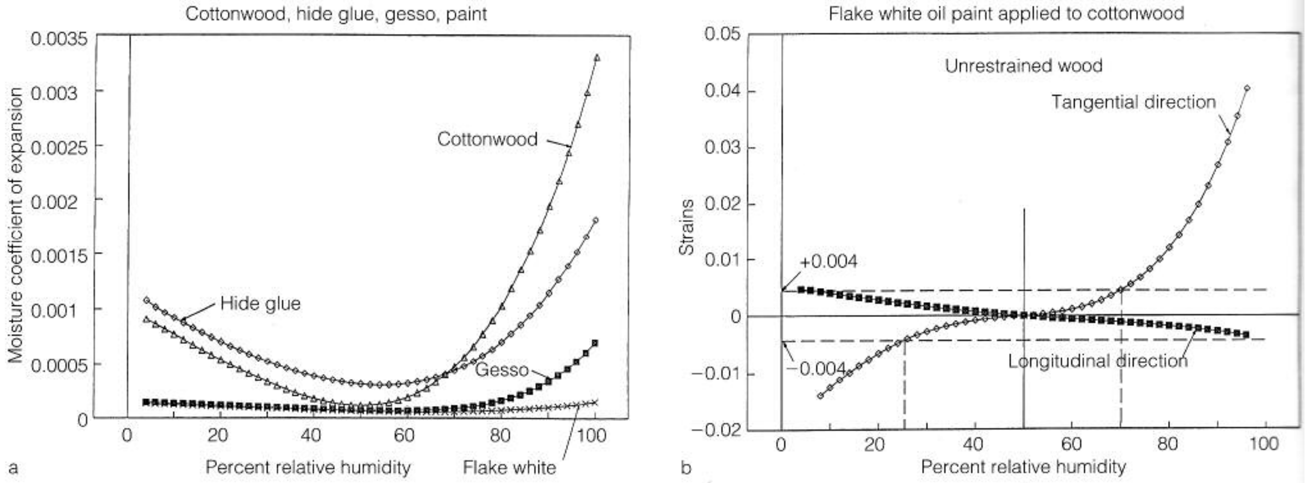


Figure 15a, b
A comparison of the moisture coefficients of expansion (a) of cottonwood, gesso, hide glue, and flake white oil paint. Equation 4 was used to determine the strain interactions (b) of flake white in oil and its cottonwood substrate in fluctuations above and below an ambient RH of 50%. At extreme low RH, the paint endures simultaneous compressive strain in the wood's tangential direction and tensile strain in the longitudinal direction. These strains are reversed at extreme high RH.

sional stability, and woods that retain paint well tend to be the most dimensionally stable. Obviously, this is the reason radially sawn boards hold paint better than tangentially sawn boards. Other than dimensional stability, density seems to be the chief factor in wood's ability to retain paint, with less dense (more porous) woods likely to be better paint substrates. This explains the tendency for paint to adhere better to earlywood in species with pronounced earlywood-latewood differences; the lower density and greater porosity of the earlywood helps it to retain paint (Marian 1967). This correlation of wood density with paint adhesion suggests that the wood-paint bond is largely mechanical.

Nonetheless, there are convincing arguments that van der Waals forces contribute significantly to the adhesion of paints to wood (Marra 1980; Salomon 1967; Kaelble 1971:45–82; Pocius 1986; Parker and Taylor 1966). Current theory suggests that the adhesion mechanisms are a combination of chemical bonding and mechanical attachment. While increasing the porosity (or surface texture) of a substrate may encourage a better mechanical grip, it is also true that more chemical bonding sites are exposed. Whether or not the adhesion is mechanical or chemical, both bonding mechanisms are seriously affected by wood's affinity for water. While moisture can seriously affect an existing paint-wood bond line, fracture testing of bonded joints suggests that the presence of moisture in the wood at the time of paint application can also seriously weaken the resulting bond (Kousky 1980).

Moisture represents one of the most important factors in the delamination of a paint film, glue size, or gesso ground. High moisture content disrupts the adhesion bond, and subsequent desiccation separates the materials physically. High moisture content in wood can result from liquid sources such as rain, condensation on walls, or groundwater from wet foundations. It may also result from a vapor source (persistent high relative humidity).

Conclusion

Fluctuations in relative humidity cause strains in the materials used to construct a cultural object. Quantifying the moisture response of each of the materials permits one to determine the allowable RH fluctuations—the RH changes an object can safely endure. In the example presented in this study (a painted panel composed of cottonwood, hide glue, gesso, and oil paint), several conditions were examined to assess the RH-related behavior. Theoretically, the hide glue size was found to be the material limiting the allowable RH fluctuations of the panel. Because of its low yield point and its great capacity for expansion in response to moisture, the glue should limit the panel to an allowable RH fluctuation of 50% ± 11%. Because glue stresses relax over time, however, the glue actually has little influence on the overall response of the panel.

The maximum allowable RH fluctuation for a particular object is ultimately determined by examining the independent RH response of each of the materials making up an object, determining the effects of the composite nature of the object on each material's response, then adopting the composite worst-case responses as the factors that truly limit the allowable RH fluctuations. Assuming that an ambient RH of 50% is desirable, the painted panel can endure maximum allowable RH fluctuations of 16% above 50% RH (the tension yield point for the tangential wood substrate) and 22% below 50% RH (the compression yield point for gesso in the wood's tangential direction). As a structure, then, the panel may endure considerable fluctuations in the environment.

Equally important, the research shows the *type* of damage most likely to occur, given a particular set of circumstances. For example, cracking is most likely in the gesso and paint layers when a panel is expanding in response to a severe increase in RH. These cracks will run parallel to the grain of the substrate (longitudinally). Cracking might also occur in the gesso and paint under extreme desiccation, but these cracks will run perpendicular to the grain of the substrate (across the grain). During severe desiccation, the gesso and paint layers may even suffer compression cleavage and ridging, but the ridges will invariably run parallel to the substrate's grain.

The research also shows that if a painted panel becomes equilibrated at an extreme high or low RH, it cannot be returned to the moderate RH regions without damage. This is because the domain of allowable RH fluctuation narrows at extreme high or low levels of ambient RH, permitting very little RH change without yielding and failure.

From the standpoint of structural stability, the optimal environmental baseline for most objects is in the middle RH region (45% to 55%), since nearly all materials experience their lowest rates of RH-induced dimensional response (i.e., their lowest moisture coefficients of expansion) in this region.

Note

1 Because the authors' tests were performed in pounds per square inch and the graphs provide data in the same units of measure, conversions to kilograms per square centimeter were not done.

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Factors Affecting the Re-Treatment of Previously Consolidated Matte Painted Wooden Objects

Eric F. Hansen and Mitchell Hearns Bishop

Consolidating porous, fragile matte paint without affecting the appearance of an object is considered by ethnographic conservators to be one of the most challenging conservation treatments. Today, due to a greater knowledge of preventive conservation and an increasing consideration of ethical issues, consolidation treatments are not so readily or routinely attempted as in the past. But many objects have been treated in the past, and conservators are often required to re-treat them. This chapter considers some factors affecting the choice of procedures and materials for re-treatment of painted wood objects in which a previous consolidation treatment produced an undesirable change in appearance or insufficiently strong consolidation.

This discussion is based on the authors' previous reviews and evaluations of treatment methods and materials. One major difficulty encountered is ascertaining what materials and methods have been used in prior conservation treatments, due in a large part to a lack of documentation. Therefore, another purpose of this chapter is to emphasize the need for a comprehensive history of the materials used in the consolidation of painted objects.

Matte, Porous Paint and Its Treatment

Some research conducted at the Getty Conservation Institute over the past five years has focused on the problems associated with the consolidation of painted wooden ethnographic objects, particularly those with matte, friable surfaces. A number of studies conducted in support of this research have been published (Hansen, Sadoff, and Lowinger 1990; Hansen et al. 1993; Hansen, Lowinger, and Sadoff 1993; Hansen and Volent 1994), along with a topical review and an extensive annotated bibliography (Hansen, Walston, and Bishop 1993) published as a special supplement to volume 30 of *Art and Archaeology Technical Abstracts* (AATA). The topical review, presented as an introduction to the AATA supplemental bibliography, includes an overview of the historical occurrence and the variety of technologies and materials used in the production of matte, porous paint, along with a discussion of analytical methods useful for the identification of the range of organic binders.

Matte, porous paint can be found on wooden objects in a wide variety of cultural contexts. This type of paint is ubiquitous in indigenous painted wooden objects from Oceania, Africa, and the Americas, including bark paintings of Australian Aborigines, masks and sculpted figures from

Africa, and painted objects from the American Southwest (such as kachina dolls). Inorganic pigments, such as red iron oxide, yellow iron oxide, calcium carbonate, and various black minerals predominate, and charcoal also was readily available as a black colorant.

Although the color palette is somewhat restricted in many examples of ethnographic painted wood objects, a plethora of organic binders is encountered due to the general use of locally available plants or animals. Thus, the types of binders encountered reflect the geographic and temporal variation of plant and animal species. Binders that have been cited repeatedly include acacia gum, pine resins, orchid juice, and animal glue (Hansen, Walston, and Bishop 1993:xix–xx).

The emphasis of this overview of matte paint requiring consolidation centered on ethnographic and archaeological objects because of the frequency with which their paint problems have been reported in the literature; however, similar problems are also found with matte paints used in architecture, folk art, modern and contemporary art, and many other forms of applied art. Some outstanding examples are Netherlandish Tüchlein distemper paintings and medieval German panel paintings, illuminated manuscripts, North American Colonial period house paints, and nineteenth- and early-twentieth-century pastel drawings.

It is interesting to note that in the first three of this last set of examples matte paint requiring consolidation can be associated with the historic use of proteinaceous binders such as animal glue (gelatin). More recently, problematic matte paint requiring consolidation is seen to be the result of the formulation of paint with an unusually low ratio of the volume of a synthetic polymer binder—such as poly(vinyl acetate)—to the volume of pigment in order to achieve a matte surface for a particular aesthetic effect. Examples are works by artists such as Yves Klein, Mark Rothko, Barnett Newman, and Ed Ruscha (Hansen, Walston, and Bishop 1993:xxvii–xxviii).

However, the primary purpose of the topical review was to assess current information on the properties of paint that is in need of consolidation and to explain how such information might be used to develop treatment strategies. The following is a brief summary of the authors' findings on the reasons for the "success" or "failure" of consolidation treatments in adequately consolidating paint while minimizing changes in appearance. It serves as an introduction to the focus of this article: factors to be considered in the re-treatment of paint whose previous consolidation altered the appearance to an unacceptable degree or failed to obtain cohesion of the paint and adhesion of the paint layer to the substrate (in this case, wood).

The friable or flaking porous condition of a painted surface is often due to an insufficient amount of binder in the paint formulation, whether from an intentional or unintentional act on the part of the artist or artisan. Paints with a high ratio of pigment to binder are described in the coating industry as a "high pigment volume concentration coating." For the purposes of this discussion, high pigment volume concentration paints have two important properties: low strength and extendability; and low gloss (a light, matte color). When a consolidant is introduced in the usual manner, through spray or brush application of a solution, void spaces are filled. While filling of void spaces binds pigment particles together, this may also result in a reduction of scattered light from air-pigment interfaces, which causes darkening. If there are localized high concentrations of consolidant at the surface, there may also be an increase in the gloss. Additionally, noticeable *tidemarks* may appear at the edge where a consolidant solution flowed, if the entire surface has not been covered.

The aim, therefore, when consolidating porous, matte paint, is to use a consolidation system that distributes the consolidant in a manner that minimizes changes in appearance by introducing the minimum quantity necessary to achieve effective cohesion of the paint and adhesion of the paint to the substrate, while maintaining long-term compatibility with the paint and support materials.

Two distinct classes of paint have been previously considered: powdering porous paint, and flaking porous paint. In the case of flaking porous paint, consolidants used to adhere flakes may wick into the flakes and cause darkening. In the AATA topical review, a schematic arrangement of possible treatment options was split into two categories based on their utility in consolidating a particular paint type (Hansen, Walston, and Bishop 1993:xlvi). The primary strategy employed for the first category—powdering paint—is to use various methods to maintain a low viscosity of the consolidant solution as a means of maximizing penetration and equal distribution. (This may be achieved by means of low volatility solvents, suppression of solvent volatility, or multiple applications of dilute solutions.) The primary strategy employed for the second category—flaking porous paint—is to use various methods that minimize penetration into a flake by an adhesive. (Methods include highly viscous solutions, spray techniques, and pretreatment with solvents immiscible with the consolidant solution.) Another strategy employed for both types of paint is to use methods that discourage leveling of the consolidant solutions, and thus promote conformity of the solid consolidant on the surface to the roughness of the paint film.

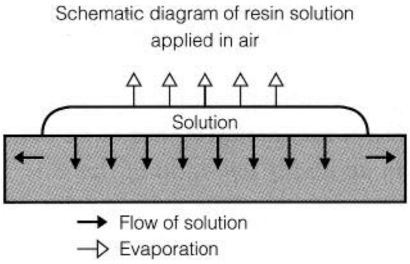

Figure 1
Schematic diagram of resin solution applied in air. The direction of solution flow is indicated by the solid black arrows; evaporation is indicated by the open arrows (after Hansen, Lowinger, and Sadoff 1993).

Thus, for high pigment volume concentration paint, "successful" treatment options are those that achieve the desired distribution of a consolidant. "Unsuccessful" treatment options are those that create unfavorable resin distribution and placement within the paint layer, which causes darkening, the formation of tidemarks, or insufficient cohesion of the pigment particles and poor adhesion to the substrate.

Figure 1 illustrates the flow of a solution into a paint layer, shown in cross section, while solvent is simultaneously evaporating from the surface. Because the concentration of the solution flowing into the paint increases with time as the solvent evaporates, the final consolidant concentration (a darker area indicates a greater concentration of resin) may be greater at the surface (Fig. 2a), causing darkening, possibly increased gloss, and low concentrations at the substrate-paint interface. By contrast, tidemarks may result from deposition of materials originating from solubilization of resins in the wood, from dirt or products of biodeterioration, or from the physical redistribution of pigment particles (particularly when there is a large particle size distribution) as a result of solution flow. However, tidemarks may also result from the deposition of consolidant. As a solution flows outward from its applied area, solvent is evaporating, increasing the concentration and viscosity of the solution with time. The result may be a resin distribution (Fig. 2b), where greater concentrations of resin at the edge result in darkening from greater filling of void spaces or from changes in surface reflection.[1]

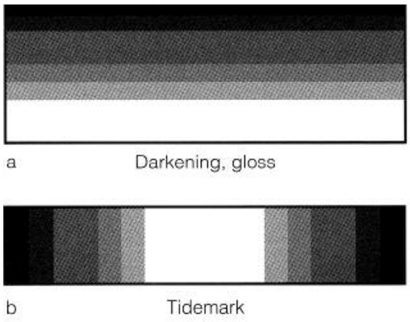

Figure 2a–b
Possible concentration profiles of resin solution applied in air, shown in cross section; darker areas indicate relatively higher concentrations of resin (after Hansen, Lowinger, and Sadoff 1993).

General Considerations in Re-Treatment

As stated earlier, the primary reason for re-treatment is that previous consolidation treatment(s) (1) resulted in an unacceptable change of the surface of the painted object (i.e., the surface was originally lighter and more matte but is now darker, grayer, or glossier); (2) failed to impart the required strength of cohesion and adhesion to prevent further paint loss; or (3) failed in both respects. The specific aim of re-treatment of matte paint is to minimize change in the original appearance of the object and to stabilize a now unstable or deteriorating surface. Although the ethical goal of using reversible treatments may have little practical applicability, the use of materials and procedures that are amenable to future manipulations (such as future solubility), whether applied in the past or in use today, allow for the possibility of re-treatment.[2]

Differences in the reflection of light between surfaces must be avoided if treated and untreated areas are to match in appearance. This phenomenon (which has long been recognized in the coatings industry), where two colors appear to match at one angle but no longer match if the viewing angle is changed, is termed *geometric metamerism* (Johnston 1967). The diagram of geometric metamerism reproduced in Figure 3 shows the effect of pigment protruding from a film on the scattering of light, and it explains why in some instances a tidemark can be detected only by rotating the plane of the paint in relation to the eye. Therefore, a further treatment, or re-treatment, that either removes excess consolidant from the surface of the paint layer or (as is generally more practical for the systems discussed) redistributes a consolidant into the desired concentration throughout the paint layer will minimize discoloration and increase cohesion and adhesion.

The most important factors affecting re-treatment of matte painted wood objects are

1. the chemical and physical nature of the consolidant, which either promotes or discourages solubilization and redistribution in the future;
2. the parameters of methods for solubilizing previous consolidants to allow removal or redistribution without disrupting or damaging a fragile paint layer;

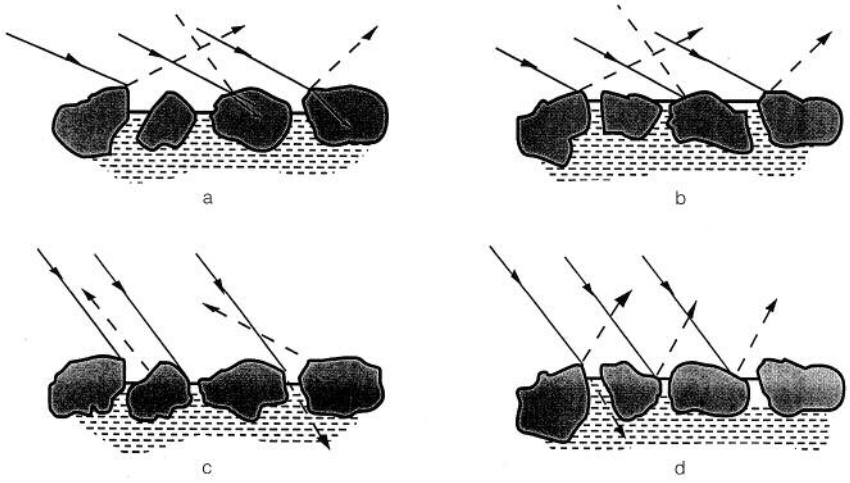

Figure 3
Geometric metamerism (after Johnston 1967). The colors of a and b will appear to match. The colors of c and d will be different.

3. the condition of the wood substrate and possible ground layer(s), and the potential effect of added solvents and materials upon the substrate and ground (both immediately and in the long run).

Evaluation of these three factors requires knowledge of past treatment—in particular, the materials used and their aging properties.

In reference to the wood substrate, it may be quite deteriorated and may have been previously consolidated prior to, or during, the consolidation of the paint. One must also consider the effects of solvents (used to deliver the consolidant) on the wood, as well as the penetration of the consolidant solution (and therefore added resin to the wood), resulting both from the original treatment(s) and from subsequent corrective re-treatment.

Conservation Materials

Identification of methods and materials used in previous treatments

When re-treating an object, an attempt is made to determine what substances may have been applied in the previous treatment. If some form of treatment documentation exists, it may be possible to find the information. Unfortunately, this is rarely the case for early treatments. If any documentation on previous treatments does exist, it may not be accessible to the conservator, or it may be sketchy and inadequate since it may date to a period prior to the codification of conservation documentation.

When it is not possible to find documentation of previous conservation materials and application methods, two alternatives for identification may be used. The first is the analytical approach. A number of methods ranging from optical microscopy to chromatographic techniques and a variety of instrumental methods may be used to determine what materials may have been applied. These analyses can be strengthened by a knowledge of materials that were *likely* to have been used during a given time period.

If no conservation documentation exists, literature from a given period can give some idea of the methods and materials that were in use. Until a definitive work on the history of conservation materials is published, it will be necessary to rely on literature such as published conservation texts and articles from the relevant period. Some of this information can be found in *Materials for Conservation* (Horie 1990) and in compilations such as *Early Advances in Conservation* (Daniels 1988). *Dégradation, conservation et restauration de l'oeuvre d'art* (Marijnissen 1967) contains an important and extensive discussion of the history of conservation that has been cited by others (Keck 1977; Keck 1984), but it has yet to be translated into English. Further sources include oral histories (such as those found in the Foundation of the American Institute for Conservation Archives or the Archives of American Art of the Smithsonian Institution).

A systematic review of materials cited in early treatises, works on artists' techniques, and early conservation literature waits to be undertaken, along with a consideration of the dates that many of the synthetic polymers were likely to have been introduced into conservation. For example, it is possible to make a comparison between the methods and materials advocated by Alfred Lucas in his publications, and the actual treatment documentation for the objects from Tutankhamen's tomb that were treated in the late 1920s. In *Antiques: Their Restoration and Preservation* (1924), Lucas describes the preparation of conservation materials he recommends. He suggests three materials for consolidation: paraffin wax, a solution of celluloid in amyl acetate or acetone, and a solution of cellulose acetate in acetone. He proposes the use of a spray atomizer or a camel-hair brush for application. Lucas also suggests a re-forming technique in the following remarks in a reference to the treatment of "black lacquer" Egyptian wooden funerary objects (1924:96):

> This is not a paint, but a kind of varnish, consisting of a natural black resin of a lacquer-like character, such as is found and used in India, China and Japan at the present day. . . . [B]eing a resin it is soluble in alcohol and acetone, and if sprayed with either of these solvents, should it show signs of flaking off, it softens at the edges and adheres again. The spraying makes the surface very glossy, but in many instances this was the original appearance.

Reference to the actual treatment records left by Lucas verifies that in fact consolidants used on painted wood objects from Tutankhamen's tomb did involve paraffin wax, celluloid solution in amyl acetate, celluloid solution in acetone, cellulose acetate in amyl acetate and acetone, and cellulose acetate in acetone (Piqué 1994). Methods of application involved pouring of the melted paraffin wax, and spraying and brushing on of the celluloid and cellulose acetate solutions. Such information should be confirmed whenever possible by analytical means before proceeding with a re-treatment program because many objects (especially those judged the most important objects in a collection) may have been given multiple treatments.

Aging and solubility of conservation materials

An abbreviated list of materials used for the consolidation of paint in the twentieth century is given in Table 1, roughly in order of their introduction. Of primary importance for this discussion is the stability of the

Table 1 Materials used for the consolidation of paint on objects

Wax
Natural products (drying oils, gums)
Gelatin
Cellulose nitrate
Cellulose acetate
Poly(vinyl acetate)
Poly(methyl methacrylate)
Poly(vinyl alcohol)
Poly(butyl methacrylate)
Acryloid B72
Poly(vinyl butyral)
Soluble nylon
Cellulose ethers
Thermosetting polymers (e.g., epoxies)

materials in reference to environmentally introduced chemical modifications that decrease solubility, such as cross-linking of polymer chains. Arguably, the most stable synthetic polymers are poly(vinyl acetates), poly(methyl methacrylate), and Acryloid B72 (an acrylic copolymer), when they are supplied as a solid resin alone or dissolved in an organic solvent. (These materials may be supplied in the form of aqueous emulsions formulated with a variety of additives; in some cases, they may have undesirable aging properties, as well as often undesirable working properties.) Paraffin wax—as opposed to a variety of plant and animal waxes processed by various methods—is also relatively resistant to oxidation; therefore, painted objects previously treated with wax might be retreatable (although wax introduces several other problems, discussed below, that may render it inappropriate for use as a consolidant).

Paint consolidated with such materials may possibly be re-treated by removal or redistribution. However, less stable materials, such as butyl methacrylate or cellulose nitrate, offer less possibility for re-treatment, depending on the severity of environmental extremes to which the consolidated objects have been exposed and the length of time that has lapsed since consolidation.

Of particular interest—and the point of this discussion—is the fact that a greater potential exists for the re-treatment of some objects treated with materials in very early time periods—for example, objects treated with poly(vinyl acetates) in the late 1930s and 1940s, or even with paraffin wax in the 1920s—than for objects treated with some of the materials introduced later—especially those consolidated in the 1960s and 1970s with soluble nylon or poly(vinyl acetate) and poly(vinyl alcohol) emulsions.

The authors have previously discussed the possibility of using thermosetting polymers in low concentrations to consolidate matte paint, based on the consideration that greater strength of consolidation could be gained by introducing smaller amounts of thermosetting resins (such as epoxies) than with thermoplastic resins, thus minimizing the potential for undesirable changes in appearance (Hansen and Agnew 1990). Additionally, it has often been considered that the solubility characteristics of aged material are of little relevance because of the very unlikely chance that a material could be removed in the future from a very fragile surface such as porous, powdering paint (Horton-James, Walston, and Zounis 1991). In light of the factors affecting re-treatment presented here, the use of thermosetting polymers should be more carefully reviewed, along with the argument that future solubility of consolidation materials is not an important working consideration.

Re-Treatment of Wood

One of the most common early treatments for consolidating painted wood objects, particularly when the wood itself was in need of consolidation, was the application of, or immersion in, molten wax (Rosen 1950). Hatchfield and Koestler (1987) used scanning electron microscopy to evaluate the microstructural appearance of archaeological wood that had been subjected to consolidation with wax, followed by subsequent removal of wax, and then a further consolidation with an acrylic resin. It was found that, even though wax removal was incomplete after successive immersions in toluene, subsequent consolidation by the addition of an acrylic resin in toluene resulted in improved strength and appearance of the wood. When a similar wood was consolidated with the acrylic resin alone, subsequent removal of the acrylic resin by solvent immersion was more complete but appeared to cause more damage. In fact, the greatest damage was observed when the wood was immersed solely in toluene without any consolidation treatment.

This study not only illustrates the possibility for re-treating painted wood objects originally treated with wax, but also suggests that residual amounts of a consolidant may reduce the amount of disruption to a fragile surface when it is re-treated, while still allowing for the introduction of a new consolidant. The presence of paint on wood considerably complicates the problems associated with consolidation, particularly when it is necessary to find a consolidant procedure or procedures for both wood and paint (systems that differ both in respect to their structure and to the materials present). Even when the wood substrate does not necessarily need consolidation, one must consider the possible effect on the wood when the paint is consolidated. The creation of a barrier coating through consolidation of the paint might affect the equilibrated moisture content of the wood at different relative humidities (i.e., a change in the dimensional response of the wood substrate with fluctuations in relative humidity might affect flaking or loss of adhesion—particularly in respect to adjacent treated and untreated areas). However, this may be of little concern because, by definition, successful treatments introduce the smallest possible amount of material, leaving the porous paint as close to the original porosity as possible to retain the object’s initial appearance.

One further factor to be considered is resin migration in wood that has been treated with a consolidant. Schniewind and Eastman (1994) found that samples of wood that had been completely saturated with a consolidant by an immersion treatment subsequently had an uneven distribution of the consolidant when dry, with some indication of a tendency to increase concentration toward the end surfaces as a result of migration of the consolidant resin with solvent evaporation. As previously mentioned, the authors have not found reverse migration to be a viable phenomenon in highly porous or powdering paint; rather, the localized surface concentrations in the paint layer are the result of lack of penetration due to solvent loss through evaporation. However, the dynamics of distribution of the consolidant in the wood substrate as a result of penetration into the wood by the consolidant used for the paint, and the subsequent evaporation of solvent through the porous paint, are not as well understood. Hence, questions persist as to how the consolidant remains distributed in the wood, and what effects this might have on the wood substrate (both in relation to the original consolidation treatment and to subsequent re-treatment methods, including those for removal, redistribution, or reintroduction of consolidants).

Examples of Removal or Redistribution of a Previously Applied Consolidant

Removal of an unwanted varnish layer on matte paint

One successful re-treatment involved the removal of an undesired coating (originally applied as varnish layers) from an easel painting that was in such a fragile state that a preconsolidation treatment was required. The varnish had added undesired gloss and darkening to the originally matte surface, and a new consolidant was added to stabilize the fragile, porous paint layer, allowing removal of a previous consolidant. Although on first consideration this example of the conservation of an easel painting may seem to be somewhat removed from the problems associated with painted wood, it should be stressed that very few treatments of paint on any substrate to remove past materials specifically to return the surface to a matte appearance are known to the authors. Of particular interest is the newly developed technique of using solvent gels to clean painted surfaces. These methods may have potential for the removal of unwanted consolidants from the paint on wood surfaces.

The Adoration of the Magi, attributed to Andrea Mategna (active in the fifteenth century), is in the collection of the J. Paul Getty Museum. In the Tüchlein technique, the medium is distemper (glue based) and results in a lucid, matte surface that was the desired aesthetic intention, as opposed to that later associated with the more saturated and glossy surface of drying-oil paints that were subsequently varnished. Over the centuries, layers of grime had built up on the thin and very porous paint surface. Varnishing to revive the surface had resulted in further darkening. The purpose of the conservation treatment was to remove the varnish, as well as the grime, and not to revarnish the painting (Rothe 1992:85).

The paint—which was porous, fragile, and showed lifting of numerous small flakes—was first consolidated with several applications of dilute aqueous gelatin solutions. All attempts to remove the varnish with organic solvent mixtures produced extensive blanching. However, preliminary tests with solvent gel formulations and procedures developed by Richard Wolbers of the University of Delaware/Winterthur Museum Art Conservation Program specifically for this application allowed varnish removal without blanching (Rothe 1994). These particular solvent gels were formulated with polymers of acrylic acid that can be used to thicken both aqueous and organic solvent solutions. Gelled solutions can be used to control solvent evaporation rate, extend contact time with the surface, and, to some extent, control the degree that a solution permeates into a surface.[3] Two solvent gel formulations were used: one formulated with acetone, water, and benzyl alcohol, and another with ethanol, xylene, water, and Triton X-100, in addition to the thickening agent. The gels were removed with Shellsol 71 (now called Shell Odorless Mineral Spirits) after application to one small section of the painting at a time. The majority (but not all) of the varnish could be removed from the entire surface in this manner.

Thus, not only in principle but in practice, it is possible in some instances to remove an unwanted consolidant from a fragile paint layer. In this case, the varnish had penetrated into the porous paint and was not completely removed because a satisfactory appearance had been achieved. The formulations of the solvent gels are mentioned not to suggest any recipe for removal of commonly encountered picture varnishes, such as dammar or mastic, but rather to underscore the fact that the blanching associated with organic solvents was diminished, presumably because the penetration of the solution into the paint was reduced. Solvent gels may be effective in the removal of any number of synthetic and natural products due to the wide choice of applicable solvents. Additionally, this example may be of interest to some ethnographic conservators in Europe, where, although the practice of using natural products such as dammar to consolidate paint is rarely encountered today, such was not the case in the past few decades (Hansen, Walston, and Bishop 1993:lvii).

Achieving satisfactory results in the removal of varnish from matte paint requires an inordinate amount of skill and experience on the part of the conservator, specifically in regard to avoiding effects such as geometric metamerism of successively treated adjacent areas of the paint (as shown in Figure 3, where a similar appearance of two paints is shown to be dependent on a similar depth of protrusion of pigment particles from the paint surfaces).

Regarding the use of solvent gels, some concern has been raised that the gels are either not totally effective against capillary action—allowing moisture penetration through cracks and into size layers (Southall 1989)—or (in the case of aqueous systems, such as resin soaps) that the residues of nonvolatile components may affect properties of paint films, such as oil paints (Erhardt and Bischoff 1993).

As stated earlier, a great majority of matte painted wood objects are ethnographic objects from Oceania (including Australia) and Africa that are executed with little paint binder and in which the use of a limited palette of inorganic pigments (particularly red and yellow iron oxide) was extensive. In many instances, there may be little potential for problems associated with the use of solvent gels or resin soaps in the re-treatment of ethnographic objects, particularly if the paint can be treated with a consolidant (such as gelatin) prior to attempts at removing a resin. However, because ethnographic objects are often produced using locally available colorants and binders, the diversity of materials encountered can be much greater than that usually found in traditional Western easel paintings. In such cases, the potential for interaction effects might increase.

Redistribution of a previously applied consolidant

The second example is that of the successful redistribution of a consolidant that had caused darkening of a light, fragile matte paint layer (formulated without the addition of a binder) when originally applied. As stated in the previous example, reasons for paint discoloration may be the uneven distribution of resin. Localized surface concentrations may add gloss or fill void spaces, causing darkening. Localized concentrations may also define the extent to which a solution flowed over and through the paint, appearing as tidemarks. Therefore, re-treatment methods that successfully redistribute the consolidant may reduce these changes in appearance, while still maintaining sufficient cohesion of the paint and adhesion to a substrate, or even improving the cohesion and adhesion.

Hansen, Lowinger, and Sadoff (1993) treated a number of wood blocks, which were painted with red iron oxide and yellow iron oxide on a white kaolin ground, in two different ways: in an atmosphere open to the environment, and in an atmosphere saturated with the solvent used to make the consolidant solutions. The pigments were applied from an aqueous slurry with no added binder, which resulted in a very powdery surface.

The blocks were treated in 1989 with 5% solutions of poly(vinyl acetate) AYAF[4] in acetone, ethanol, toluene, and xylene, and a similar concentration of Acryloid B72 in the same solvents, along with a solution in diethylbenzene. The center area of the blocks were left untreated so that a visual comparison of an untreated and a treated area could be evaluated, along with the formation of tidemarks. Color images (Hansen, Lowinger, and Sadoff 1993:9) illustrated that the darkening that occurred in an open atmosphere (when acetone, toluene, and xylene were used as solvents) was not apparent when the consolidant solutions were applied in a saturated atmosphere.

In late 1994, working on the assumption that these two polymers should be soluble in the future, the blocks were re-treated by spot treatments with solvent alone. Acetone could not be delivered to the previously consolidated painted surface by brush techniques without removing pigment; however, acetone could be delivered from an eyedropper without disturbing the surface. The discolored areas of four blocks, two previously treated with Acryloid B72 in toluene and two previously treated with poly(vinyl acetate) in acetone, were treated by spot application of acetone in a vapor-saturated atmosphere. (When treated with one to three applications of acetone in an open atmosphere, the discoloration was undiminished.)

One block of each consolidant type was placed in a glove bag containing open pools of acetone for thirty minutes, then treated while still in this environment. Discoloration of red and yellow areas disappeared, but was still quite noticeable in white areas. However, two other blocks that had remained in this environment for two hours were treated with three applications of acetone; after removing these blocks from the glove bag (allowing residual solvent to evaporate), it was not possible to visually discern where the consolidant had been applied. It was, however, possible to distinguish the areas that had been previously consolidated from the central unconsolidated area because pigment could be removed from the center by touching it lightly with the finger. Previously consolidated areas re-treated with acetone still retained their resistance to pigment removal.

This example illustrates the possibility for re-treatment of a previously consolidated object, either to eliminate darkening or to remove tidemarks that are assumed to be the result of uneven consolidant distribution, if the original consolidant is not expected to become insoluble. Such a method is less complicated than attempted removal of a consolidant, and in this instance no preconsolidation was required. However, it has been noted (Hansen and Volent 1994) that when solvent evaporation is retarded, and the solvent allowed to remain in contact for longer periods of time, greater potential exists for solvent interactions with sensitive materials present in the paint layer, ground, or substrate. Solvent interaction tests done in an open atmosphere may not be adequate to readily determine suitable solvent systems for use in a saturated atmosphere; solvent interaction tests must be done under the conditions in which they will be used.

Conclusion

The re-treatment of matte painted wood objects requires both a knowledge of the materials and methods that were used in the past, and an understanding of the reasons why the original treatment may have been unsuccessful. For many objects treated before—or without—the systematic documentation of procedures and materials, ascertaining specific re-treatment options may be difficult even if requisite analytical support is available. Another requirement is a knowledge of how the materials (including solvents) were used and how compatible the properties of the consolidated paint layer are with the wood substrate, an area that has not been extensively studied.

Where specific documentation does not exist, a general knowledge of available materials and techniques during specific time periods and in specific geographic areas may be of help. A definitive summary or review does not now exist, but an initial compilation could begin with teaching texts and "handbooks" for conservation treatments, articles describing case studies, treatment reports on file in museums or other institutions, and also with oral histories. In addition to identification of the materials that have been used, designing re-treatment strategies requires a knowledge of the expected aging characteristics of individual materials, particularly in regard to solubility.

For those materials that remain soluble, and when the discoloration or lack of consolidation is a result of uneven consolidant distribution (and not of "staining" or other solvent interactions, including physical disruption of the surface due to the consolidation procedure), several possibilities for re-treatment exist. One possibility is removal of a previous consolidant, which may require another consolidation procedure prior to removal. Another possibility is to remove excess consolidant while leaving enough residual consolidant to maintain cohesion and adhesion. It may also be possible to redistribute the consolidant in a manner that reduces the final concentration but does not contribute to glossiness or darkening.

In regard to the first possibility, newly developed techniques for cleaning varnishes may have great potential for the treatment of painted wood objects. Regarding the redistribution of previous consolidants, the use of a saturated vapor atmosphere to inhibit solvent volatility has been discussed (and may be particularly important for the removal of tidemarks). However, other methods that work on a similar principle (such as using a low-volatility solvent) also have potential.

It has been suggested in the conservation literature that future solubility is not a requirement when the original paint surface is highly fragile, because it is not likely that a resin can be removed from such a surface. It has also been suggested that thermosetting resins may have possible applications because they might be used in very small quantities. Since it is now apparent that re-treatment of fragile surfaces may be possible, these approaches should be reconsidered.

Acknowledgments

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Notes

1 Alternative explanations for darkening encountered in the conservation literature have been based on either the differences in the refractive indexes of pigment particles and consolidants, or *reverse migration* of a consolidant solution to the surface as a result of solvent evaporation. The reasons these phenomena have been considered by the authors to have minimal relevance in the consolidation of porous paint have been extensively discussed by Hansen, Lowinger, and Sadoff (1993) and Hansen, Walston, and Bishop (1993:xlviii–xlix).

2 See Appelbaum (1987:67) for a more general discussion of internal consolidation and re-treatability in relation to the concept of irreversible treatments.

3 See Stavroudis (1990) for more detailed comments on the use and formulation of gelled solvents.

4 PVA AYAF is a molecular weight grade of poly(vinyl acetate).

Materials and Suppliers

Acryloid B72, Rohm and Haas Co., Independence Mall Street, Philadelphia, PA 19105.

PVA AYAF, Conservation Materials, Ltd., 100 Standing Rock Circle, Reno, NV 89511.

Shell Odorless Mineral Spirits, A. G. Layne, 4578 Brazil St., Los Angeles, CA 90039.

Triton X-100, Sigma Chemical Company, P.O. Box 14508, St. Louis, MO 63178.

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The Ultrasonic Mister

Applications in the Consolidation of Powdery Paint on Wooden Artifacts

Stefan Michalski, Carole Dignard, Lori van Handel, and David Arnold

Powdery matte paint is a paint layer with little or no binder between the pigment particles; it can therefore transfer easily when there is contact with another surface, or it can detach if it is submitted to vibration. In the consolidation treatment of powdery paints, the aim is to improve both the cohesion of the pigment particles and their adhesion to the substrate, while minimizing the effect of the consolidation on the appearance of the paint layer (Michalski and Dignard 1997).

Often, improvements in handling, storage, packing, and transportation procedures offer a viable alternative to consolidation treatment (Guillemard and Renard 1992). However, there can be instances when the choice is either consolidation or acceptance of paint loss—for example, when a powdery object cannot be physically secured to prevent losses from normal building vibration, air movements, or vertical orientation. Static charge also can cause losses. In those rare instances where there is lack of control over access, handling, or dust protection, consolidation may be the safest preservation option. If consolidation is required, ultrasonic misting, with its advantages and limitations, should be considered as an option. This chapter will present a discussion of the method, tests performed on powdery painted wood samples, and use of the treatment method for two wooden objects of very different provenance and history. Construction of the ultrasonic mister will be described, in the hope that other conservators will experiment further with this new technique.

The Ultrasonic Mister: General Description

To atomize a consolidant solution into a mist, a dilute solution (for example, 0.5% Acryloid B72 in ethanol; 0.5% gelatin in water; or 0.25% methyl cellulose, 400 centipoises, in water) is placed in a bottle positioned over the ultrasound oscillator of an ultrasonic humidifier (Fig. 1). An air pump connected to the bottle forces the mist out through tubing to a nozzle and onto the object. Some of the mist will penetrate into the porous layer, but some will bounce off and could potentially deposit on adjacent areas of the object. To avoid this, the treatment is performed in a fume hood; alternatively, a local extraction system using a vacuum cleaner and voltage regulator can be used to suck up excess mist. Figure 1 shows the ultrasonic mister, as originally developed by Michalski,[1] and Figures 2 and 3 show details of the handpiece and various nozzles. Complete details on the equipment, setup, and method are given in the appendix to this article.

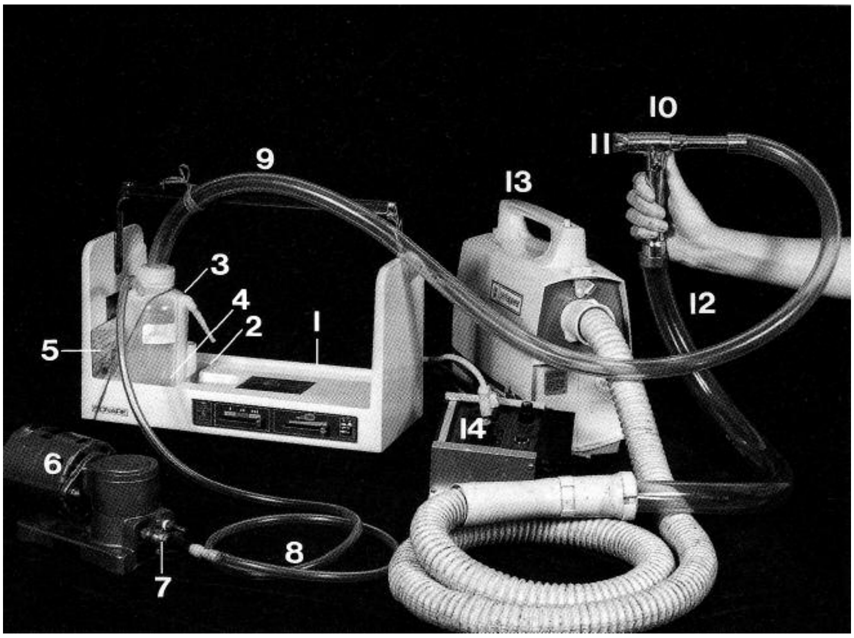

Figure 1
The ultrasonic mister: (1) ultrasonic humidifier; (2) water in the reservoir; (3) low-density polyethylene squeeze bottle, "Boston" type; (4) dilute consolidant in bottle; (5) wooden retainer to stabilize bottle; (6) air pump; (7) valve to control quantity and velocity of mist; (8) tube carrying air from pump into bottle; (9) tube carrying mist from bottle to nozzle; (10) handpiece; (11) copper pipe slot nozzle, out of which mist is projected; (12) tube carrying extracted excess mist; (13) vacuum cleaner extracting excess mist; (14) speed control for extraction force.

When misting a powdery paint, the consolidant solution must penetrate the full thickness of the pigment layer down to the substrate; otherwise, incomplete consolidation will result. If too much solution is applied, it could dry on the surface and cause skinning or glossiness and may also hinder further applications. The mist flow can be reduced or increased by using the air-pump valve. The flow remains as narrow as the aperture of the nozzle, so it can be directed with precision. The nozzle

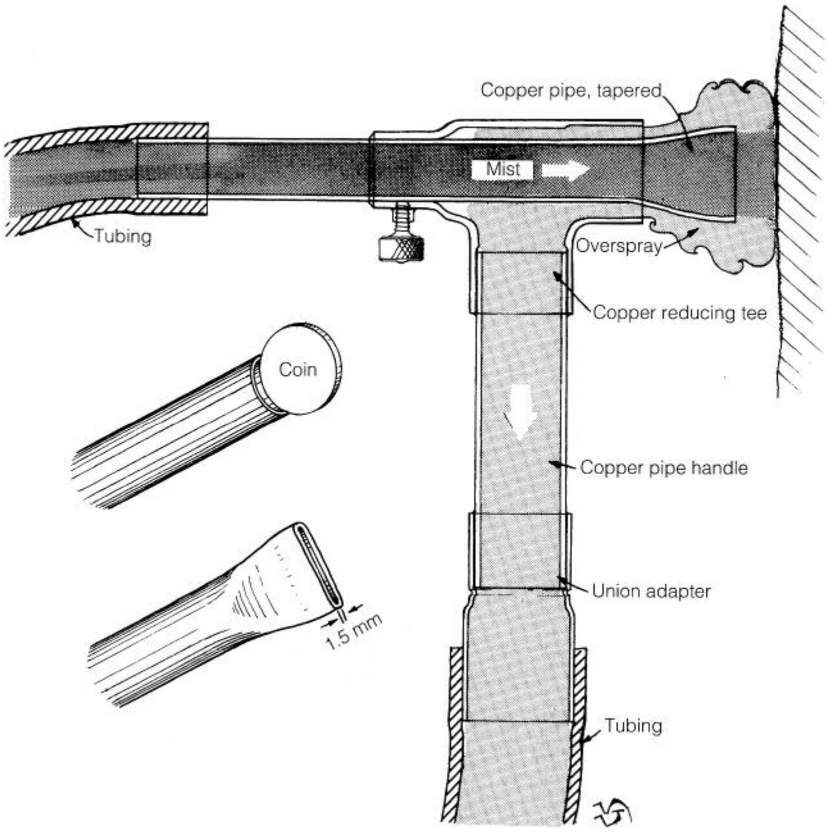

Figure 2
The ultrasonic mister handpiece (Fig. 1, no. 10), consisting of a copper pipe slot nozzle, a copper reducing tee, and a handle. Insets: a coin is inserted in the copper pipe and pinched in a vise to create a slot nozzle.

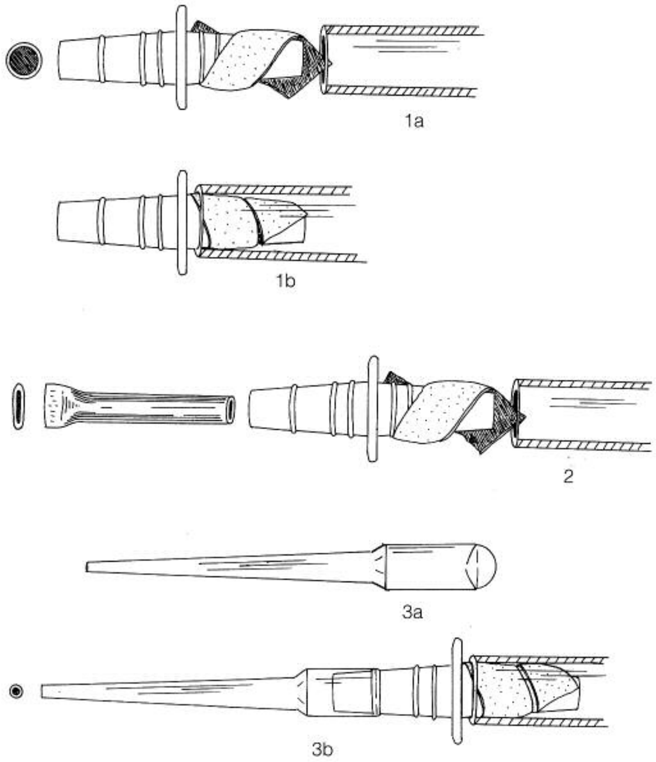

Figure 3
Various nozzle configurations: (1) quick-disconnect fitting (dimensions 6.4–7.9 mm) with (a) blotter paper wrapped around it, and (b) inserted into tubing; (2) Nalgene quick-disconnect configuration with addition of miniature copper tube (6.4 mm outside diameter); (3) disposable, plastic pipette, (a) before incorporation and (b) after it has had the end cut off and been added to Nalgene quick-disconnect configuration.

must be held within a few millimeters of the object and kept stationary for as long as the mist is seen to wick into the paint layer (it will wick until it fills all the pores, to the bottom of the layer). During preliminary testing, penetration to the bottom of the paint layer was verified by misting various consolidant solutions onto pigments applied to a transparent glass substrate (Michalski and Dignard 1997). When the surface being misted is thoroughly wetted, a shiny appearance indicates that the porous layer is saturated with solution. The nozzle should then be moved to the next area. When the solvent evaporates, it will leave only a small quantity of solid consolidant distributed within the pores. Consolidation using this device may require several applications of the dilute solution.

General Problems Encountered in Consolidation

Conservators have often encountered poor results with consolidation methods for powdery pigments—for example: (1) darkening, glossiness, or skinning due to poor penetration; (2) darkening due to color saturation; and (3) smearing. These problems are often linked to the fact that too much consolidant is being used, at concentrations that are too high. In this respect, the application technique is important. Ultrasonic misting has some advantages over other techniques such as pneumatic spraying and drop applications. (The use of vapor-saturated environments is an option independent of application technique [Hansen, Lowinger, and Sadoff 1993].) Also, the choice of both the resin and the solvent are important factors, as they can affect viscosity, wetting, evaporation rate, strength of bonding, and degree of color change (Michalski and Dignard 1997).

Lack of penetration

Lack of penetration is the most common problem in attempts at consolidation. The effects are darkening, transparency, glossiness, or skinning.

Problems with penetration can occur if the consolidant is too viscous to penetrate the small capillaries between the pigment particles, if the solvent evaporates faster than the solution's wicking time, if wetting is poor, or simply if too much consolidant is applied and it pools on the surface. The last point is a serious drawback to both drop application and pneumatic spraying, as the amount of consolidant delivered cannot be finely controlled. The minimum used in these techniques is one macroscopic drop, which for some paint layers can be too much at once. Depending on the consolidant penetration rate in the paint layer and on the evaporation rate of the solvent, a drop may take too long to penetrate and may end up drying on the paint surface. (Consolidation within vapor-saturated environments is a means of eliminating evaporation so that even complete wicking can take place in due course.)

Ultrasonic misting is a different approach, whereby dilute solutions of low viscosity are gradually applied locally and in small, controllable quantities; pooling can thus be avoided. Furthermore, because the mist is applied very close to the object, little evaporation takes place during application; the droplets are delivered in an airstream saturated with solvent, at a concentration very close to the concentration of the original solution. In contrast, pneumatic spray drops undergo rapid evaporation in the ambient air before reaching the object, to the point of becoming too viscous to penetrate.

Darkening

Darkening due to color saturation occurs when the addition of a consolidant within a porous layer reduces the void volume, and thus also reduces the air-solid interfaces that scatter light. Colored pigments become more saturated. If the pigment has a refractive index well over that of the consolidant (for most consolidants, it is about 1.5), then at least the pigment remains fairly opaque. Pigments with an index of refraction near that of the consolidant will not only saturate in color, but they will gain transparency.[2] If very little original binder is present, however (as should be the case if the surface is powdery), a significant color change can be avoided if a consolidant is applied sparingly and it is evenly distributed.

In contrast to pneumatic spraying and drop application, ultrasonic misting allows application of a flow of very small droplets of consolidant in controllable quantities. By treating the object with successive applications of a mist of dilute consolidant, the treatment can be assessed gradually, and consolidation can be stopped before the surface color becomes noticeably different.

Smearing

Smearing can occur with both pneumatic spraying and drop application because of the size and velocity of the drops hitting a delicate, powdery surface. Excess solution on the surface can also run, which can displace particles. Ultrasonic mist droplets are two to one hundred times smaller than pneumatic spray droplets.[3] Mist droplets typically range from 1 to 5 μm in diameter, comparable to average pigment particle sizes, which range from 0.1 to 10 μm. The mist velocity can be fine-tuned by a valve on the air pump, down to a minimum flow.

Precision of application is also greater with ultrasonic misting. The stream of mist comes straight out of the fine nozzle, with little flare, allowing precise control as to where the consolidant is applied; with pneumatic spraying or drop application, the minimum surface area that can be consolidated is much larger.

Limitations of the Ultrasonic Mister

While the ultrasonic mister can reduce or control the three problems noted, the following can be limitations or shortcomings to its use:

1. The machine must be assembled and fine-tuned before being operative.
2. Drops may accumulate in the tubing and nozzle and could fall on the object if the nozzle is directed downward. Blotter liners can control most of this problem (Fig. 3).
3. Since the solutions are misted in narrow bands, there can be an overlap in application of consolidant if the first band is dry when the adjacent one is applied, thus doubling the consolidant; there is concern that this may cause a pattern of lines on the object. As much as possible, adjacent bands should be butted when wet to avoid this problem.
4. The process of consolidation is relatively slow. This is a consequence of the slow delivery of solution and of the incremental application of dilute solutions.
5. Ultrasonic misting is generally not effective as a treatment for flaking, cleaving, or cupping paint because the concentration of the consolidant is inadequate to bridge the gap between flake and substrate. Also, the flake may cup on drying. Even with a very slow mist velocity, it is difficult to avoid dislodging flakes; however, tiny flakes can be consolidated by misting, as in the case of flaking gouaches (Weidner 1993; Dignard et al. 1997). Paint that is both flaky and powdery can be delicately misted to increase cohesion and to relax the shape of flakes before they are repositioned and fixed to the substrate by other means.

The limitations are discussed further in the following case studies.

Tests Performed on Painted Wood Samples

The following observations were gathered from ultrasonic misting tests, using aqueous solutions of gelatin, methyl cellulose, or solutions of Acryloid B72 in ethanol. The tests were performed on various dry pigments that had been applied as a slurry with no binder onto wood planks.

1. Individual pigments have different bonding requirements. For example, the pigment particles of green earth, red ochre, and raw umber formed cohesive layers even though no binder was present; therefore, in practice, such pigments may not need consolidation. Others such as calcium carbonate, chrome yellow, ivory black, and ultramarine were found to be much more powdery. Similarly, the improvement in bonding for a given consolidant can vary from pigment to pigment. Each pigment requires a treatment tailored to its physical cohesive strength and its propensity to change color when the consolidant is applied.

2. Solvents alone can change the color of some pigments. Distilled water applied on green earth and red ochre darkened their surfaces, while ethanol on ultramarine caused lightening of the surface (Michalski and Dignard 1997).
3. Natural resins in unaged wood can migrate and stain when the painted wood is wetted during treatment. Consolidation tests using 0.5% Acryloid B72 in denatured ethanol, applied on new pine planks painted with various pigments, caused the wood resins to migrate and stain the pigment surfaces in dark streaks. This problem is expected with solvent-based consolidants and on all softwoods, especially if new. For water-based consolidants, resin migration was rarer, but it did occur in one instance.
4. End grain will wick up the consolidant more quickly than will radial or tangential surfaces, which makes it difficult to know when enough consolidant has been applied. It may be necessary to establish the speed of application on radial or tangential sections, and then to retain this speed on end-grain surfaces.
5. The problem of dust on a powdery paint is a concern. Dust was simulated by sprinkling the painted wood samples with fine dust from a vacuum cleaner, and exposing them to thirty minutes of cigarette smoke in a chamber. Misting did not reveal any dirt migration, such as tidemarks; however, in practice, the authors still view dirt and dust as potential problems to the consolidation process and to the final visual appearance of the paint. Preliminary tests on the object are essential.
6. Mist is applied to an object in adjacent bands; where the bands overlap, the quantity of consolidant will be double; therefore, the color change could be more significant. To avoid this, each band is butted closely while avoiding overlap and, most important, the band is applied before all the solvent in the previous band evaporates. The wet consolidant can then wick into and blend with adjacent areas. Also, successive coats of consolidant are applied in bands at right angles to those of the previous coat. In practice, overlap was found to be a problem mostly after four applications of ethanol-based solutions (0.5% Acryloid B72), which evaporate faster than aqueous solutions.
7. Brushstrokes, which form a pattern of long, raised ridges on some painted surfaces, often need additional consolidation because of their greater thickness. They can be selectively treated, using a narrow slot nozzle designed to focus the mist stream along their length. Striping or other superimposed decoration may also require further misting, with care taken to prevent the mist stream from flooding adjacent areas of the base coat.

Case Studies

Egyptian painted coffin panel

This polychromed panel is one of six that are a part of a Middle Kingdom, Twelfth Dynasty coffin excavated around 1915 at the Deir el-Bersheh site on the east bank of the Nile about 175 miles south of Cairo, and now in the collection of the Museum of Fine Arts, Boston (Fig. 4). The panel

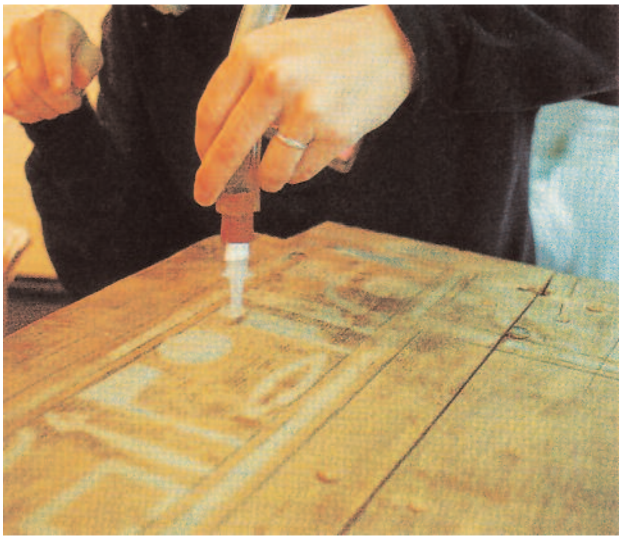

Figure 4
Consolidation of Egyptian painted coffin panel (Middle Kingdom, Dynasty 12) using the ultrasonic mister. Notice the Nalgene quick-disconnect nozzle and the blotter between the nozzle and the tubing. Museum of Fine Arts, Boston (acc. no. 1915-5-532).

serves as an end piece and was detached from the rest of the coffin during excavation and subsequent shipping. Coarse calcium carbonate gesso fills knot holes and joints, and the panel is covered with an ochre-tinted calcium carbonate ground. The painted surface is a faint band of polychromy that follows the contour of three sides of the coffin end: Egyptian blue hieroglyphs are outlined in white, and there are traces of orpiment in negative spaces of the design. Black and white border and outline the design.

Condition

The Lebanese cedar substrate consists of four thick boards that are fitted together with wooden pegs, tenons, and bands of copper. One plank is split, and the piece is detached from the rest. There are many areas of abrasion and loss to the calcium carbonate ground. The panel was stored, transported, and treated in a horizontal orientation to avoid any further loss of pigment. Prior to treatment, most areas of the painted design were poorly bound and extremely powdery. In several areas, pigment particles had previously been displaced, but the design was generally intact and legible. The Egyptian blue pigment was the most powdery and was easily transferred with surface contact. The orpiment, which is of large particle size, and the ochre ground were equally fragile. The white was relatively well bound; however, microscopic examination revealed a minute craquelure pattern. The black appeared to be fairly well bound to itself but was detached completely from the substrate. Dirt, dust, and fiber particles covered most of the surface.

Treatment considerations and objectives

The coffin will eventually be reassembled for exhibition; the end-piece panel will be displayed in its original vertical position. Due to the fragility of the decorative layer, consolidation was the primary treatment procedure. Prior to consolidation, surface dirt and fibers were removed with tweezers and the touch of a small, soft, damp sable brush.

Technical analyses to identify binding media used in the polychromy were performed on samples by the staff of the Department of Objects Conservation and Scientific Research at the Museum of Fine Arts, Boston. It was possible to distinguish minute quantities of an animal binder using high-performance liquid chromatography (HPLC), following a technique described by Halpine (1992).

Since staining of the ground and/or wood substrate is a frequent side effect when using aqueous consolidants (Hatchfield 1988), and since earlier tests indicated that staining by migration of wood resins through the pigment layer was also a threat when using solvent-borne consolidants, solvent tests were performed on non-accessioned, painted cedar fragments of similar coffins provided by the museum's Department of Ancient Egyptian, Nubian, and Near Eastern Art. Water, denatured ethanol, toluene, and acetone were applied by dropper to the surface of both bare and painted wood and allowed to evaporate. These tests indicated that, in this case, water stains were the biggest problem. A solution of 0.5% Acryloid B72 in 97% ethanol and 3% toluene was tested and chosen as the least interfering consolidant.

Consolidation

The panel was the first object treated by the authors—van Handel and Dignard—using the ultrasonic mister, and several modifications to the mister's original setup and method were found most practical. For instance, although the copper nozzle was used to consolidate broad areas of design, the Nalgene quick-disconnect nozzle (Figs. 3 and 4) was most often used because it allowed smaller local applications and could be finely directed with the fingertips. To prevent drops from forming inside the nozzle, blotter paper was inserted into the space between the quick-disconnect fitting and the tubing (Fig. 3). The blotter paper strip was changed at regular intervals; several nozzles prelined with blotter paper were used in succession to speed up the treatment. The overspray extraction unit, consisting of the reducing tee tubing, and vacuum cleaner, shown in Figure 1 (see also the appendix to this article), was not used because treatment took place in a fume hood. The consolidant was applied following the design of the object, or following the wood grain when it could be seen through the paint. In total, four coats of Acryloid B72 were applied; no color change could be perceived, and there was no evidence of lines caused by overlapping bands. Bonding was improved but it was still weak, especially in the thicker layers of the coarse Egyptian blue pigment; however, pigment particles could no longer be displaced through surface contact.

Pennsylvania-German low post bed

This painted single bed is unique because of its decorative footboard, seen in Figure 5. While similar motifs are commonly associated with early Pennsylvania chests, it is exceedingly rare to find a bed with such a decorative treatment. The painted Fraktur script includes the name of the bed's owner, Katarina Barbara Luckenbillin, and the date, 1789. Colors in the scribed design areas were applied directly to the bare wood, and the surrounding blue was carefully painted around them. The pigments include Prussian blue, lead white, and cinnabar. The rest of the bed parts were painted with a mixture of lead white and Prussian blue.

Condition

The bed parts are composed of both white pine (*Pinus strobus*) and tulip poplar (*Liriodendron tulipifera*), and include headboard and footboard assemblies and two side rails. For many years, the headboard and

Figure 5
Footboard of Pennsylvania-German low post bed, 1789, with painted design. Philadelphia Museum of Art (acc. no. 1992-10-1).

footboard had been stored on end, each with a post in direct contact with an earthen floor. Both posts became rotted and suffered some deformation and/or loss of material as a result. Prior to treatment, the paint was generally very unstable. Most areas were powdery and some areas had lost all of the paint. Thickly applied paint in the designs was especially friable. The pine boards have suffered paint losses directly over latewood portions of the wood growth rings.

Treatment began with the consolidation of the powdery paint. Once the surface was stabilized, the disfigured turnings of the posts and other losses could be restored, and areas of major paint loss could be color-compensated. The bed is now reassembled and stands in the permanent Pennsylvania-German exhibition at the Philadelphia Museum of Art.

Technical analysis

Technical analyses to identify binding media and to confirm pigment identification were performed in the Philadelphia Museum's analytical laboratory. All paint samples prepared for binder analysis with Fourier-transform infrared (FT-IR) spectroscopy were too lean to be conclusive, but the infrared spectra indicated the possible presence of a proteinaceous binder (animal glue, or possibly casein).

Besides ultrasonic misting, two other treatment options were considered. Pneumatic spray application with Acryloid B72 in diethylbenzene (Welsh 1980) was rejected as much for its lingering noxious smell as for a concern that the high-velocity output of a spray gun might dislodge paint particles. Also, a vapor-saturated atmosphere (Hansen, Lowinger, and Sadoff 1993) was rejected because consolidation within a bag was judged to be too difficult to perform and assess practically; there was also lack of laboratory space.

Consolidation

An aqueous consolidant was chosen for the pine headboard and footboard to avoid resin staining. Tests performed on samples of painted tulip poplar plaques indicated that the solution would perform equally well over the tulip poplar substrates. Some good results have been reported on producing a mist from a 0.5% solution of Dow Methocel A4C methyl cellulose in water (Dignard et al. 1997); however, consistent results could not be obtained with the Philadelphia Museum apparatus at this concentration, which is almost too viscous to mist. A 0.25% solution was generally used, although some success was experienced using concentrations as high as 0.375%.

Several modifications to the original Canadian Conservation Institute–designed apparatus were made. A pinhole was made in the shoulder of the consolidant bottle so the fluid level could be easily maintained with periodic additions by syringe of dilute consolidant. The air-input tube was modified, as shown in Figure 6 and described in the appendix under the heading "Modifying the plastic bottle." This modification is intended to direct the incoming air around the inner walls of the bottle and thus minimize disturbance of the rising consolidant mist. For convenience, a burette stand and a pair of adjustable clamps were used to independently hold both the output tube and the neck of the consolidant bottle in place. An overspray collector was fabricated, but it was not used because a good-quality voltage regulator was not available. The inexpensive one used for the treatment caused the vacuum cleaner to surge, resulting in an inconsistent, and thus unsatisfactory, performance. Despite this omission, no color change or banding could be perceived when consolidation was completed. Because of the low concentration used, eight coats of methyl cellulose (0.25%) were applied, approximately equivalent to the quantity of consolidant in one application of 2.0%. No color change or signs of overlapping can be perceived by eye.

Conclusion

The cohesion of pigment particles has been significantly improved with the ultrasonic misting technique, even in the design areas where pigments were applied thickly.

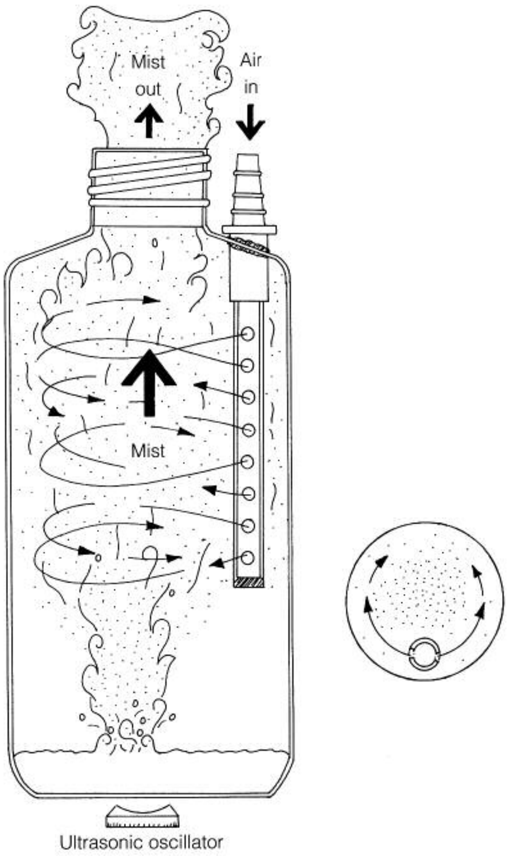

Figure 6
The air-input assemblage inserted into the shoulder hole in the LDPE bottle. The quick-disconnect fitting is hotmelted to the bottle. Inset: as viewed from above, showing air movement created by opposing 3–5 mm holes in tube.

Consolidation through pneumatic spraying, vapor-atmosphere, or drop applications was not possible in the case studies of two wooden objects with powdery painted surfaces; but ultrasonic misting was successful. The equipment is relatively easy to assemble and use, and the method offers several advantages over other application methods: small droplet size; low mist velocity; a narrow, easily directed nozzle and stream of mist; and control of the quantity and concentration of consolidant applied. Incremental applications of a dilute, nonviscous solution of consolidant mist allow one to find a compromise between minimum color change and maximum bonding.

Acknowledgments

The authors acknowledge the assistance of many colleagues: Bob Barclay, who helped to make the original mister handpiece; Janet Mason, Carl Schlichting, Anita Henry, Charles Hett, Martha Segal, Lisa Mibach, David Lee, Denis Guillemard, Julie Dennin, and Nancy Odegaard, who communicated their comments on ultrasonic misting. Thanks also to Peter Newlands, Gregory Young, Bob Barclay, Robyn Douglas, Sherry Guild, and Wanda McWilliams at the Canadian Conservation Institute; Caroline Marchand at the Canadian Museum of Civilization; and Susanne Gänsicke at the Boston Museum of Fine Arts, who experimented, or assisted with, the mister for various treatments. Appreciation is extended to Carl Schlichting, who drew some of the illustrations; Beth Price and Richard Newman for analytical services; and Arthur Beale, David DeMuzio, Tom Stone, and the Andrew W. Mellon Foundation, who facilitated this cooperative effort.

Appendix: Ultrasonic Mister Equipment, Setup, and Method

The following is a detailed procedure for adapting a household humidifier for use as an ultrasonic mister (Fig. 1). Except for the voltage regulator, all the equipment is available from hardware stores, aquarium supply shops, and plastics distributors (see Materials and Suppliers). If a conservation lab owns a voltage regulator, a vacuum cleaner, and an air pump (or has a compressed air source), the approximate cost is Can$125 (US$100). Only the handpiece may need to be custom-built, using simple materials and tools.

It is important to note recent research, undertaken subsequent to the development of the modified ultrasonic humidifier, which explored two devices used in respiratory therapy that adapt more readily for use as consolidant delivery systems (Arnold 1996:102). When tested over the entire range of mist-producing consolidant concentrations, a pneumatic nebulizer and an ultrasonic nebulizer both produced mist particles with mean diameters of 0.7 to 1.1 µm. Both devices also generated mists using solutions at significantly higher concentrations than the converted humidifiers, and the ultrasonic nebulizer demonstrated superior control and precision. Although the pneumatic nebulizer is the least expensive means for producing consolidant mists (if a compressed air source is available), an ultrasonic humidifier, modified according to the following directions, remains a less expensive alternative to an ultrasonic nebulizer.

Equipment and setup

Modifying the plastic bottle

Numbers shown in parentheses in the following paragraphs refer to Figure 1. A plastic squeeze bottle (3) for containing the consolidant solution must be modified for use within the reservoir of the ultrasonic humidifier. Large, low-density polyethylene (LDPE) “Boston” design squeeze bottles allow the ultrasound to pass through and create a mist of consolidant inside the bottle (PVC and high-density polyethylene bottles do not work). Cut or melt a hole in the lid of the bottle, and one on the shoulder, to fit the lengths of tubing leading to the handpiece/nozzle and pump, respectively.

An optional step is to modify the air-input tube (8) and to secure it to the bottle in the following manner: cut a 10 cm length of 6 mm internal diameter (ID) tube, and seal it at one end with hot-melt glue; then, cut a series of 3–5 mm holes in opposing sides of the tube (about six holes, spaced evenly along the length of the tube). Insert the unsealed end of the tube into a Nalgene quick-disconnect fitting that has been hotmelted to the bottle. Figure 6 shows this modified air-input assemblage inserted into the shoulder hole in the LDPE bottle.

Small air pump and tubing

An air pump (6) of at least 200 l hr^{-1} output without resistance, is connected by a 6 mm ID tube (8) to the bottle. The pump blows air into the bottle and forces the mist out into the nozzle tubing. The air pump may be connected to a variable transformer to regulate the air flow, or simply to a valve (7) to control the quantity and velocity of mist coming out. A standard compressed air source can be used.

Nozzle and tube

The mist travels along clear 16 mm ID tubing (9) to the nozzle. Tubing walls 4 mm thick should be used to avoid kinks or sharp bends. Because some of the consolidant will collect on the tubing walls and drain to the lowest point, try to keep the tubing lower than the nozzle to ensure that the solution does not inadvertently drain out of the nozzle onto the object.

A circular nozzle (Fig. 3) was found useful in practice because it is small and lightweight, and it can be finely handled to direct the mist where required. Nalgene quick-disconnect fittings (6.4–7.9 mm) were found to be most useful. Nozzles with low-angle tapers are best, as they reduce the formation of droplets. Use blotters, as shown in Figure 4 and as discussed in the treatment of the Egyptian panel, earlier.

A nozzle with a slot aperture (11) can be useful for applying an even, uniform quantity of consolidant when it is moved across a surface. A slot aperture 20 mm long by 1.5 mm wide was found to be most practical. To make this, follow these steps:

1. Taper or flatten one end of a 15 cm length of copper pipe with a 13 mm ID (Fig. 2), by holding a large coin partway into one end of the copper pipe while squeezing the pipe in a vise until the pipe walls re-form against the coin faces. The taper angle should not be too abrupt or droplets of consolidant will form inside the nozzle.
2. Make the faces of the aperture parallel to each other.
3. File the inside lips of the aperture to make them smooth (burrs or irregularities will act as nucleation sites for droplets, which may fall on the object).

4. Droplets will still form; therefore avoid pointing the nozzle downward, and frequently wick up any droplets forming by slipping a blotter in the nozzle aperture during use.

Copper pipe was chosen to make the slot nozzle because it is readily available and easily worked. After two years of intermittent use, no corrosion products formed on the prototype, which was rinsed and blow-dried after use. If copper impurities are a concern, use polycarbonate (Lexan), which is inert and transparent, but more difficult to form. Since the nozzle is easily replaced, other nozzle shapes and sizes can be created to suit particular needs, as seen in Figure 3.

Overspray extraction system

Some of the mist that reaches the object is absorbed into the object, and some stays in the deflected airstream as overspray. There was concern that overspray settling on adjacent surfaces might cause glossiness or hinder future penetration of the consolidant. There was also concern regarding the serious health risk that consolidant mist can pose, even with aqueous solutions. An overspray extraction system using a copper reducing tee and handle (10), a vacuum cleaner (13), and speed control (14) is most effective. Unlike a fume hood, it is a mobile system, which works locally and allows the suction to be varied as needed. However, with flammable solvents, the fume hood must be used instead, since a flammable mist drawn through a vacuum cleaner motor can ignite and cause an explosion. With toxic, nonflammable solutions, the reducing tee extractor can be used in the fume hood.

Reducing tee and handle. Assemble the reducing tee and handle (10) as follows:

1. Slip a copper reducing tee over the nozzle, with the larger diameter encircling the nozzle aperture.
2. Connect the bottom part of the tee, through a pipe (the handle) and tubing, to the vacuum cleaner. To do this, screw or solder a 19 mm copper pipe to the tee to provide a handle, and a 19–25 mm reducing coupler to the bottom of the handle to fit the 25 mm ID vacuum cleaner tubing.
3. Screw the tee to the nozzle before use.

When assembled as shown in Figure 2, a 6 mm vacuum collar encircles the nozzle aperture, capturing overspray from the periphery of the nozzle. The force of the extraction is adjusted by changing the distance between the collar and the tip of the nozzle, or by the vacuum speed control.

Vacuum cleaner, tubing, and speed control. The vacuum cleaner (13) provides the force of extraction. Connective tubing (12) of the same diameter as the vacuum cleaner's normal hose, usually at least 25 mm, should be used. A wet/dry vacuum cleaner is recommended, if available. Normal vacuum cleaner suction must usually be reduced to less than 10%, using either a controlled leak in the vacuum cleaner hose or tubing, or a variable speed control (14)—either a solid-state speed control or a variable transformer of appropriate amperage.

Method

Filling the reservoir and installing the plastic bottle

Add enough tap water to the recessed reservoir (2) of the humidifier to maintain the manufacturer's recommended operating level once the bottle is in place. Center the bottle (3) over the ultrasound oscillator, making certain no air bubbles are trapped beneath it. Secure the bottle with a wooden retainer (5), a twist tie, or a metal clamp and support.

Quantity and concentration of solution in the bottle

For water-based solutions, use the same height of consolidant in the bottle as in the water reservoir (i.e., 2–3 cm of solution in the bottle). Ethanol-based consolidants require between 0.5 and 1 cm of solution, with the bottle raised 1 cm from the oscillator, using a ring. Viscous solutions will not mist. Use very dilute solutions—for example, 0.5% aqueous or ethanol solutions.

Adjusting the stream of mist

Keeping the humidifier controls at their maximum levels, adjust the mist velocity, using the valve on the pump (7), so that the mist moves fast enough to impinge on the surface, but not so fast as to disturb loose particles.

Distance between object and nozzle

Hold the nozzle 5 mm or less from the surface and perpendicular to it. At this distance, the mist remains in a narrow jet, and no significant evaporation of solvent will occur before it hits the object.

Quantity of solution applied during each application

As mentioned earlier, the consolidant solution must penetrate the full thickness of the pigment layer being consolidated, through to the substrate; otherwise, incomplete consolidation will occur. If too much solution is applied, it will dry on the surface and may cause skinning or gloss and hinder further applications. Control the application as follows:

1. Adjust the stream of mist to a slow, delicate flow.
2. Keep the nozzle stationary above the object and apply the solution as long as it is seen to wick into the paint layer. The solution will wick until it fills all the pores within the layer, to the bottom of the layer.
3. When the wet surface being misted begins to appear shiny, indicating that the porous layer is now saturated with solution, move the nozzle away to let the consolidated layer dry: the solvent will evaporate, leaving only a small quantity of solid consolidant distributed within the pores. *Do not move the nozzle back and forth in the rapid sweeping motion typical of spray painting: this will cause skinning.* In practice, all of this occurs fairly rapidly, so the nozzle is slowly moved across the object as the region under the nozzle is observed to be saturated but not yet flooded.
4. Butt adjacent bands closely, before they dry, and avoid overlap.

Effective concentration of consolidant applied to the object
Complete the treatment as follows:

1. Allow the solvent to evaporate completely between each application.
2. Assess the color change and improvement in bonding between each application.
3. Apply successive coats of consolidant to increase, in increments, the final consolidant concentration on the object.

Notes

1 This technique was originally demonstrated by Michalski in a workshop titled "The Consolidation of Painted Ethnographic Objects," presented in 1990 by the Getty Conservation Institute, Marina del Rey, California.

2 For example, a thin layer of ultramarine on a white background will change from a pale opaque blue to a transparent deep blue glaze. White calcium carbonate on a dark surface will change from an opaque white to a dark gray, or disappear completely.

3 For ultrasonic nebulizers, the drop range is between 1 and 5 μm (Shoh 1979:469). For common air (pneumatic) sprayers, mean drop size between 10 and 100 μm, with the smaller droplets, is possible only at very high air flows and pressures (Fair 1979:479). For airless (hydraulic) sprayers or spritzers, the drop range is 200–4000 μm (Fair 1984).

Materials and Suppliers

From local hardware stores: ultrasonic humidifier; wet/dry vacuum cleaner; 6 mm (¼ in.) vinyl tube; screw and bolt.

From aquarium or pet shops: Wisa 120 air pump (120 l hr^{-1} in water, 200 l hr^{-1} in air); air-pump valve.

From plumbing supply stores: copper pipe, 13 mm (½ in.) in diameter; copper pipe, 19 mm (¾ in.) in diameter; copper T piece, 19 mm × 19mm × 13 mm (¾ in. × ¾ in. × ½ in.).

From plastics supply houses: flexible PVC tubing, 25 mm (1 in.) ID and 28 mm (1⅛ in.) outside diameter (OD); clear flexible PVC tubing, 16 mm (⅝ in.) ID and 24 mm (15⁄16 in.) OD; low-density polyethylene ("Boston") bottle, 500 ml size; high-density polyethylene Nalgene quick-disconnect pieces, 6.4–7.9 mm (¼–⅚ in.) or various sizes; Lexan tube, 13 mm (½ in.) ID and 16 mm (⅝ in.) OD.

From electric supply houses: Powerstat speed control 3PN116B voltage regulator, single phase, 120 V, 50/60 Hz, 10 A—from Electro Sonic Inc., 1100 Gordon Baker Road, Willowdale, Ontario, Canada, M2H 3B3; or

Staco variable transformer no. 09-521-110, 120 V, 0–120–140 V, 50/60 Hz, 10 A; or Minitrol Power Control no. 11-472-75, 120 V, 13 A—both from Fisher Scientific, 2761 Walnut Ave., Tustin, CA 92781.

From chemical suppliers: Acryloid B72 acrylic resin (methyl acrylate/ethyl methacrylate copolymer), Rohm and Haas, Independence Mall West, Philadelphia, PA 191052; or Manse Road, West Hill, Ontario, Canada M1E 3T9.

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Poly(2-Ethyl-2-Oxazoline): A New Conservation Consolidant

Richard C. Wolbers, Mary McGinn, and Deborah Duerbeck

The Dow Chemical Company received, in 1977, the first of a series of patents for a new tertiary amide polymeric material based on the monomer 2-ethyl-2-oxazoline.[1] It was quickly discovered that the new polymer exhibited some unusual physical and chemical properties. Poly(2-ethyl-2-oxazoline) (PEOX) was soluble in a wide range of solvents, including water, and it was miscible with a wide range of common polymeric materials. Even in blends with other miscible polymers, single glass transition temperatures (the temperature at which the mechanical properties of a glassy material begin to change) were often observed. As a miscible polymer, it was expected that PEOX might enhance the adhesion of other polymers to a wider range of substrate materials. PEOX also had the potential for being heat sealable, with a glass transition temperature of 55 °C. As a water-soluble material with a heat-sealing capacity, such applications as a biodegradable adhesive for packaging or cartons were initially suggested. The first comprehensive review of the physical and chemical properties of PEOX was published by Chiu and coworkers (Chiu, Thill, and Fairchok 1986).

Summary of Properties of PEOX

The structure of PEOX is given in Figure 1. The polymer is essentially prepared by a cationic ring-opening polymerization of the monomer 2-ethyl-2-oxazoline. Chiu and coworkers (1986) used this synthetic route to produce the polymer in quantities sufficient to physically and chemically characterize its properties. They measured the density of the polymer as 1.14 g cm^{-3}. The refractive index was determined to be the same as glass (1.520). Melts of the polymer appeared to be relatively shear stable; that is, when subjected to shearing or mechanical forces for prolonged periods, very little breakdown in viscosity was noted. This resistance to viscosity loss or breakdown seemed to suggest an unusual mechanical stability. When compared to other water-soluble polymers, solutions of PEOX appeared to be several orders of magnitude less viscous at comparable concentrations than the other polymers tested. PEOX was also found to be thermally quite stable (up to 380 °C in air, 400 °C in nitrogen). As mentioned above, the solubility data for the polymer suggested that a broad range of solvents would be useful solubilizers; solubility data from Chiu and coworkers is reproduced in Table 1 for convenience.

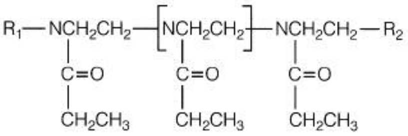


Figure 1
The structure of poly(2-ethyl-2-oxazoline) (PEOX), trade named Aquazol.

Dow licensed production of the polymer in the early 1990s to Polymer Chemistry Innovations, who began producing it on a limited

Table 1 Solubility of Aquazol

δ (solubility parameter)	Solvent	Solubility
7.0	n-pentane	p
7.4	diethy ether	p
7.8	diisobutyl ketone	p
8.5	n-butyl acetate	p
8.9	toluene	p
9.3	perchloroethylene	p
9.3	dibutyl phthalate	p
9.3	chloroform	s
9.3	methyl ethyl ketone	s
9.5	ethylhexanol	s
9.6	methyl acetate	s
9.7	methylene chloride	s
9.9	acetone	s
10.0	dioxane	s
11.9	acetonitrile	s
12.7	nitromethane	s
12.7	ethanol	s
13.3	propylene carbonate	s
14.4	methanol	s
23.4	water	s

p = solubility of 2% or less by weight
s = solubility of 25% or more by weight
(from Chiu, Thill, and Fairchok 1986)

scale under the trade name Aquazol, in molecular weight ranges of 50 K, 200 K, and 500 K. To date, the polymer has found limited use as a hot-melt adhesive, and in some pressure-sensitive adhesive preparations. Additionally, it has gained acceptance as a greenware binder because of its clean burnout and nonionic nature. Aquazol has been approved by the FDA for use as an indirect food additive (adhesive).

Scope of the Present Study

The wide solubility range of PEOX is a potentially attractive property for conservation uses. The possibilities of application or removal of residue using a wide variety of solvents could be a valuable asset in the treatment of complex painted surfaces. Another attractive feature of the polymer is its heat sealability, which takes place in a temperature range (around 55 °C) close to that of a number of synthetic resins currently in use in conservation (BEVA 371, poly[butyl methacrylate], poly[vinyl acetate] mixtures, etc.). As a conservation material, however, questions as to its stability needed to be raised and evaluated experimentally. Was it stable to light aging? Was its appearance altered on exposure? Did it cross-link? Would it oxidize or be difficult to redissolve in time? What were its handling properties? The purpose of this article is to present the results of accelerated light-aging tests on the resin and to describe a series of test applications to a selected group of painted objects.

Materials and Methods

Sample preparation and light aging prior to testing

Samples of Aquazol 50 and Aquazol 500 were obtained as dry granular powders.[2] Solutions of each were prepared in deionized water (20% w/v, given as number of grams of solute per 100 ml of solvent), and poured over standard borosilicate glass plates (6.35 cm × 20.32 cm). The cast films obtained on drying were measured for average thickness with a caliper micrometer and determined to be 0.036 mm for Aquazol 500, and 0.037 mm ± 0.005 mm for Aquazol 50 (ten repeat measures). The films were allowed to dry for one week, and then were exposed to accelerated light aging in an Atlas Ci35 Weather-Ometer at a constant 50% relative humidity, 63 °C black panel temperature, under a water-cooled xenon arc lamp (fitted with a type S borosilicate inner filter and a soda lime glass outer filter). The cast films were exposed to a 72-hour lamp "on" period, followed by a 72-hour dark period, and then a second 72-hour lamp "on" period. The total radiant exposure received was estimated to be 280 kJ m^{-2}. This value was estimated from the discoloration (yellowing) of a standard polystyrene chip,[3] measured as the increase in the *b* value, or coordinate, of its CIE L*a*b color measurement before and after exposure against a standard white background plate, as per SAE J1960 and SAE J1885, industry standard methods for exposure and dosimetry of automotive exterior and interior materials, using water-cooled, xenon-arc-controlled irradiance devices.[4] SAE J1960 specifies a constant irradiance of 0.55 W m^{-2} at a wavelength of 340 nm (1.1 W m^{-2} at 420 nm) for the xenon arc lamp. Under normal museum exposures (light sources that produce irradiances at UV wavelengths of 75 mW m^{-2} or less) (Thomson 1978:23), the total exposure obtained was equivalent to approximately twenty-four years of natural aging.

Size-exclusion chromatography

As an aid in determining the molecular weights of the Aquazol 50 and 500 materials before and after light aging, a size-exclusion chromatography (SEC) experiment was performed. A column was constructed from a 50 ml glass burette fitted with a Teflon stopcock. A small piece of glass wool was inserted in the burette above the stopcock to retain the exclusion gel slurry. A slurry of Sepharose CL-6B (20% in ethanol) was introduced into the column. Once the column was filled with the slurry, a 1 l reservoir of 0.1 M phosphate buffer (pH 7.5) was attached to the column head, and the buffer allowed to flow by gravity through the column until approximately three column volumes of buffer were passed through it. The flow rate of buffer eluent through the column after this initial equilibration stage was 0.36 ml min^{-1}. The void volume of the column (V^o) was determined by measuring the amount of eluent that passed through the column until a standard molecular weight compound, Blue Dextran—which was completely excluded because of its high molecular weight—was eluted. The elution volumes (V^e) for three other standard compounds were determined, to calibrate the column.[5] Column fractions were monitored at a wavelength of 280 nm for the standard compounds with a UV–VIS spec-

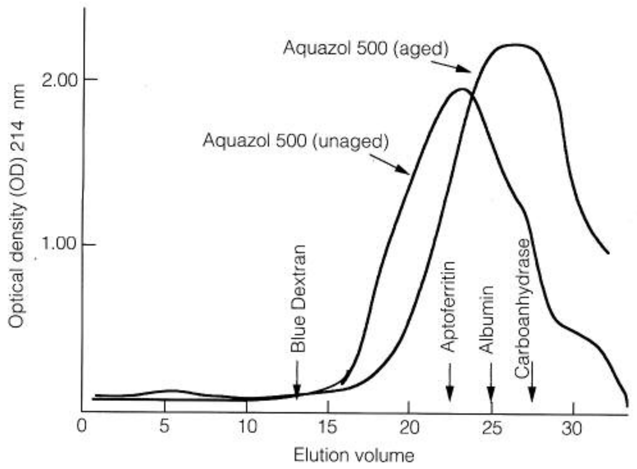


Figure 2
Size exclusion chromatography of aged and unaged samples of Aquazol 500 on a column filled with 500 Sepharose CL-6B.

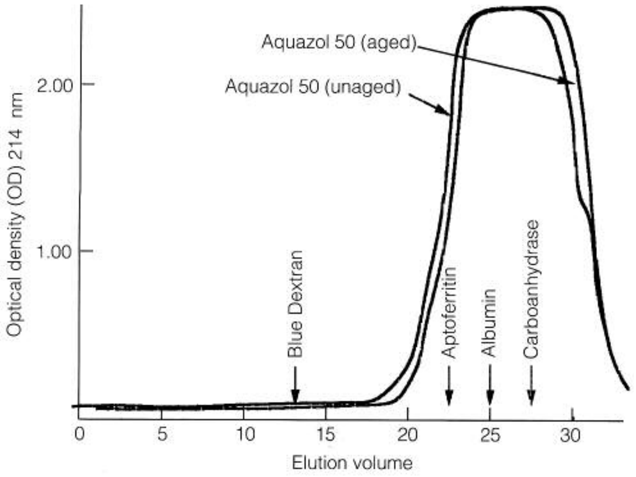


Figure 3
Size exclusion chromatography of aged and unaged samples of Aquazol 50 on a column filled with 500 Sepharose CL-6B.

trophotometer.[6] The expected linear plot of log MW versus (V^e/V^o) was obtained for the standard molecular weight compounds used, thereby confirming that the column was properly calibrated.

Samples of both aged and unaged Aquazol 50 and Aquazol 500 were applied to the calibrated column to determine their molecular weights by exclusion chromatography. The Aquazol samples were applied in 2 ml aliquots of the phosphate buffer and glycerol mixture used for the standard compounds, and at a concentration of 5 mg ml^{-1}. Both of the light-aged Aquazol materials appeared to be completely re-soluble in the phosphate buffer and glycerol mixture used for loading them on the SEC column. Their elution volumns (V^e) were tracked at a wavelength of 218 nm (the optimal absorption wavelength) with the UV–VIS spectrophotometer. The data obtained are plotted in Figures 2 and 3 for the unaged and aged Aquazol 500 and 50 materials, respectively, as a function of absorbance (optical density units) and of V^e.

pH measurements

The pH measurements were performed on 5% (w/v) aqueous solutions of Aquazol 500 and Aquazol 50 in deionized and degassed water, before and after light aging, to check the neutrality of the starting materials in each case and to determine if light aging had produced any ionizable functional groups on the polymers.[7] The pH meter and electrode were recalibrated with standard buffer solutions prior to measurement of the test solutions. A blank pH measurement was initially determined for the deionized and degassed water that was used to dissolve the tested polymers. Three replicate pH measurements were made for each polymer solution type. Results of the pH measurements appear in Table 2.

Table 2 Results of pH measurements of Aquazol 50 and 500 before and after light aging

	pH before light aging	pH after light aging
Aquazol 50	6.4 (0.1)	6.2 (0.1)
Aquazol 500	6.4 (0.1)	6.2 (0.1)

Values in parentheses indicate standard deviations of measurements.

Thermogravimetric analysis and differential thermal analysis

In an effort to repeat the observations of Chiu and coworkers regarding the thermal stability of Aquazol 500, and as a way of evaluating its thermal stability after light aging, thermogravimetric analysis (TGA) was performed on three 1 mg samples of unaged and light-aged Aquazol 500, under nitrogen in glass capillary tubes, at a heating rate of 10 °C min^{-1}. Results are plotted in Figure 4, as average curves derived from the replicate runs, for the unaged and light-aged samples.[8]

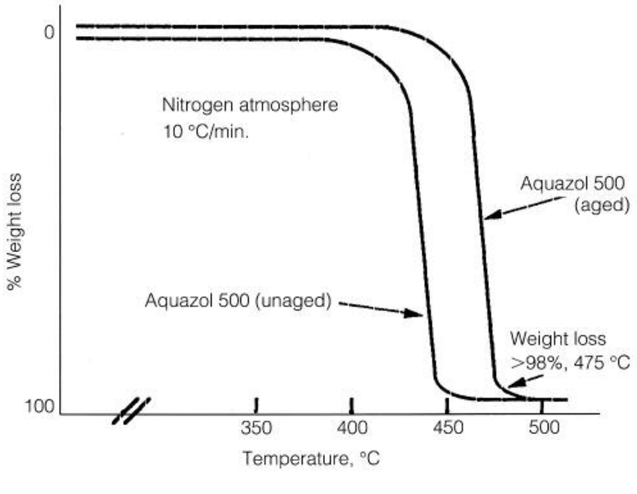
Differential thermal analysis (DTA) was performed on samples of unaged and light-aged Aquazol 50 and 500, in an effort to look for changes in such fundamental polymer characteristics as glass transition temperature (T_g), degrees of crystallinity, or specific decomposition patterns or pathways. By looking at any endothermic or exothermic transitions that might occur at elevated temperatures, fundamental structural changes in the polymer after light aging might be inferred. Observed increases in T_g, for instance, might suggest increased cross-linking in the polymer after light aging. DTA was performed on approximately 1 mg samples of aged and unaged Aquazol 500 and Aquazol 50, in triplicate, in glass capillary tubes under nitrogen, using a DuPont 900 thermal analyzer—at a heating rate of 5 °C min^{-1}—and standard Chromel Alumel Thermocouples—with the instrument calibrated against gypsum. Results are plotted in Figures 5 and 6, as average curves derived from the replicate runs, for unaged and light-aged samples.

Re-solubilization tests

In an effort to evaluate the rates of re-solubilizing cast films of Aquazol 50 and 500 in various solvents before and after light aging, a novel solubility test was devised. Typically, past re-solubilization tests have been largely empirical; that is, solvent-laden cotton swabs were rolled or rubbed over test films by hand, and the re-solubilization rates of tested materials judged subjectively. In an attempt to avoid the subjective pitfalls of past testing methods (differential solvent evaporation rates, hand-pressure variations in rubbing or rolling, etc.), a new method was devised, as follows: casting troughs were made from lengths of poly(terfluoroethylene) bar stock (3.1 mm × 25.4 mm × 30.5 cm), each notched lengthwise with a V-shaped trough approximately 1 mm in aperture and 1 mm in depth. One end of each trough was blocked with microcrystalline wax; the opposite end of the bar stock was elevated 12–13 mm to form a slanted, closed-end groove. Each trough was then loaded with a single line of borosilicate

Figure 4

Thermogravimetric analysis of Aquazol 500 before and after aging.



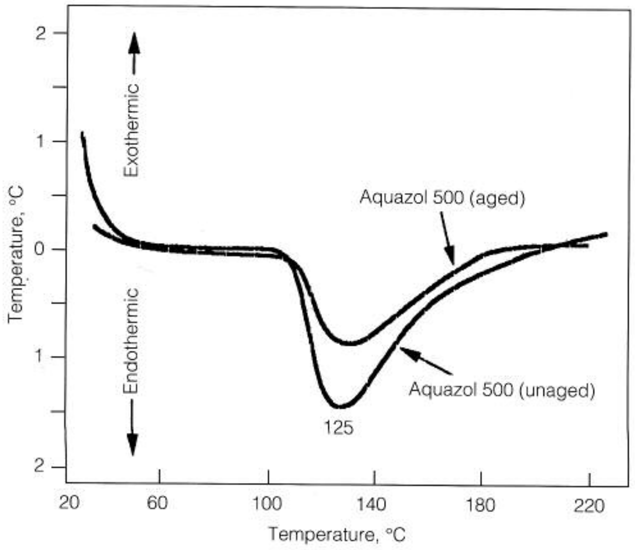


Figure 5
Differential thermal analysis of aged and unaged samples of Aquazol 500.

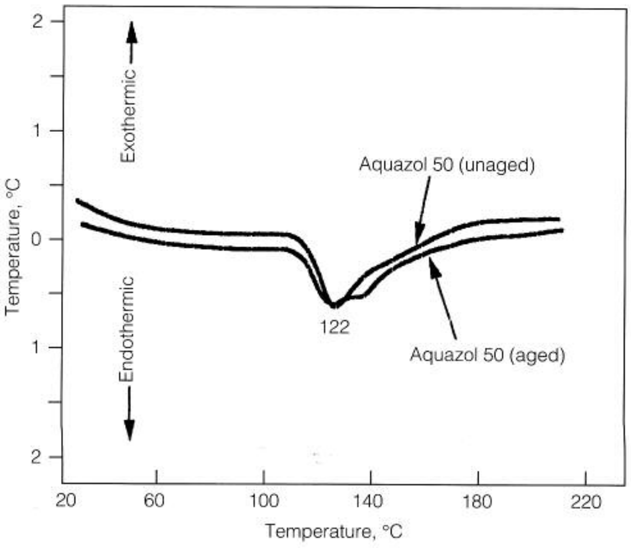


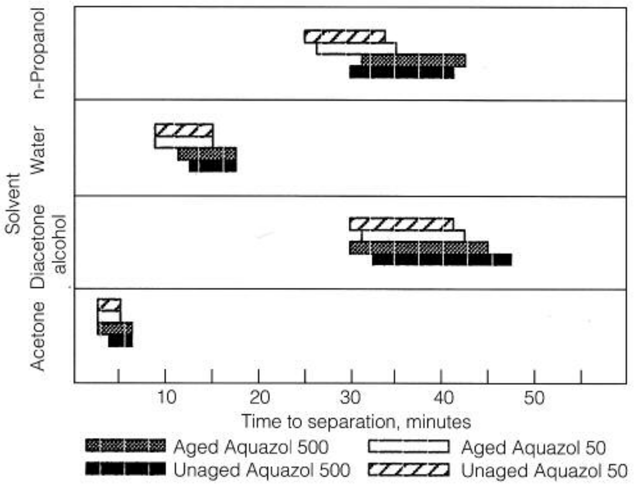
Figure 6
Differential thermal analysis of aged and unaged samples of Aquazol 50.

glass spheres[9] that just touched one another and were held in contact by gravity. Over the lines of arranged glass beads, 10% solutions of unaged Aquazol 50 and 500 in deionized water were applied with a disposable pipette. Excess solution was drawn off by pipette, leaving a thin, uniform coating on the beads and at the points of contact between them. The coated beads were allowed to air dry on the slanted forms for three days at 25 °C to form "strings" of adhered beads. Half of the coated strings of beads were light aged under the conditions described earlier in "Sample preparation and light aging prior to testing."

For this test, pairs of coated, unaged or light-aged beads were removed from the longer strings, and were then ready to be used as test substrates in re-solubilization tests. A pair of beads would be held in a given solvent solution (100 ml reagent grade) by clamping one bead of the pair with a pair of Rankin forceps.[10] The suspended pair of beads was immersed in the test solvent and stirred on a magnetic stirring plate[11] at a speed setting of 4; the time they took to separate (one bead falling free from the clamped bead) was recorded in minutes. The test was repeated five times in each solvent for these time-to-separation measurements. Results for both unaged and light-aged Aquazol 50 and Aquazol 500 are summarized in Figure 7, in the form of histographs indicating the time-to-separation ranges for the five tests in each category.

Viscosity measurements

A Brookfield viscometer, model LVT, equipped with spindle no. 1, was used to measure the viscosity of test Aquazol solutions at 25 °C, before and after light aging. Viscosity measurements were performed to help determine the molecular weights of the light-aged Aquazols; using the Mark-Houwink equation previously determined for the polymer by Chiu and coworkers (1986), viscosities that were measured after light aging could be useful to suggest whether the polymers were increasing or decreasing in molecular weight after aging. Only two replicates were performed during these viscosity measurements on each polymer sample, due to the relatively large amounts of material required to make up the test solutions. Results are recorded in Figure 8 and Table 3.



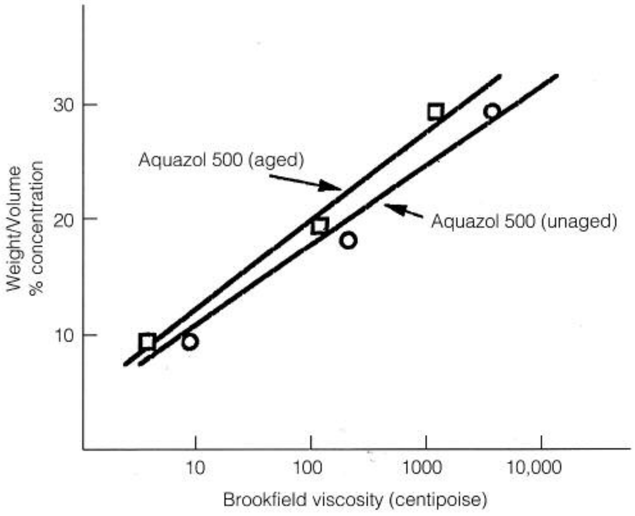


Figure 7
Time to separation (resolubility) of Aquazol 50 and 500, both aged and unaged.

Figure 8
Aqueous solution viscosity vs. concentration of Aquazol 500 at 25 °C, before (○) and after (□) aging.

Table 3 Estimated molecular weights of Aquazol preparations before and after aging

Sample	Intrinsic viscosity [η] dl/g	Calculated molecular weight
Aquazol 50	0.3	50,000
Aquazol 50 (aged)	0.27	44,000
Aquazol 500	0.83	300,000
Aquazol 500 (aged)	0.68	210,000

Estimates were made using the Mark-Houwink equation derived by Chiu and coworkers: $[\eta] = 6.5 \times 10^{-4} M^{0.56}$.

Color measurements

Color measurements were performed on cast films of Aquazol 50 and 500 (described in the section "Sample preparation and light aging prior to testing") on glass backed with a standard white reference plate, before and after light aging in the Weather-Ometer. Color coordinate values (five replicate measures for each resin type) were measured and calculated using a Minolta C100 Chroma Meter (a tristimulus colorimeter) equipped with a DP100 microprocessor unit. Results are summarized in Table 4.

Infrared spectroscopy

In an effort to establish whether any gross chemical changes might have occurred in the polymer preparations after light aging (e.g., oxidation, deamination, etc.), Fourier-transform infrared (FT-IR) spectra were obtained for Aquazol 50 and Aquazol 500 before and after light aging.[12] The unaged and light-aged Aquazol samples were dissolved in chloroform and cast as thin films before their spectra were run.

Tensile strength measurements

Tensile strength measurements were performed on 25 mm–wide strips of cast films (0.450 mm ± 0.005 mm, five replicates) of 20% solutions of Aquazol 500 in acetone, on silicone release Mylar sheets.[13] Samples of the films were preconditioned in closed chambers over saturated salt solutions (KOH, $MgCl_2 \cdot 6H_2O$, and $NaNO_3$) for twenty-four hours, prior to testing, to equilibrate them to a variety of relative humidity (RH) conditions. The samples were removed from their conditioning chambers and immediately tested on the Scott apparatus in an environmentally controlled testing room conditioned to ASTM standard D1776 (70 °F, 65% RH).[14] Results are summarized in Table 5.

Results and Discussion

The test films were remarkably unchanged visually after light aging. Color measurements before and after light aging indicated no significant difference in terms of discoloration (Table 4). Specifically, no positive increases were noted in the measured *b* coordinate values (L*a*b notation)

Table 4 Color measurements on aged and unaged films of Aquazol 50 and 500 on glass

Sample	CIE L*a*b color coordinates		
	L	a	b
Aquazol 500	96.97 (.09)	20.97 (.05)	−8.60 (.02)
Aquazol 500 (aged)	96.87 (.09)	20.80 (.02)	−8.34 (.05)
Aquazol 50	96.71 (.15)	20.97 (.05)	−8.63 (.08)
Aquazol 50 (aged)	96.49 (.10)	20.93 (.03)	−8.27 (.08)

Standard deviations for each value are given in parentheses.

Table 5 Tensile strength data for Aquazol 500 conditioned to various RH levels

RH	Elongation to yield ϵ_y (%)	Yield stress σ_y (kg m^{-1})	Elongation to break ϵ_B (%)	Breaking stress σ_B (kg m^{-1})
8%	12	44.62	380	53.57
33%	25	40.17	450	49.10
73%	50	17.85	550	26.78

that would indicate yellowing of the materials. (By way of reference, the standard polystyrene chip that was included as a dosimetry measuring device in this experiment *did* yellow on light aging, to a pale transparent yellow color, equivalent to 13.5 *b* coordinate units.) The exposed samples remained essentially transparent. Again, any decrease in transparency would have been indicated by a decrease in the measured *L* values over the standard white plates. It should be noted, however, that after light aging, the Aquazol 50 samples exhibited a distinct surface-texture change, a kind of surface reticulation ("orange peel"), that was not present on the unaged films. This effect was attributed to local water spotting and a concomitant re-forming of the test film surfaces during light aging (water droplets were blown up onto the test films from a water reservoir bath at the bottom of the aging chamber by a circulating fan in the chamber).

The FT-IR data obtained on the Aquazol samples before and after light aging were essentially unchanged. As expected for a complex aliphatic tertiary amide, major absorption bands were noted at 2998 cm^{-1}, 2950 cm^{-1}, and 2890 cm^{-1} (−CH2−, −CH3), along with a sharp band at 1660 cm^{-1} (tertiary amide). Several minor bands (1250 cm^{-1}, 1209 cm^{-1}, and 769 cm^{-1}) were present as well, suggesting the presence of an aromatic sulfonic acid, or sulfonate ester. Chiu and coworkers indicated that methyl tosylate was used in the original preparation method for PEOX as an initiator (in ratios as high as 200:1, monomer to initiator), and it may be that this material was utilized in the preparation of the present polymeric materials. It is conceivable that methyl tosylate or its hydrolysis product, p-toluene sulfonic acid, could account for the added absorption bands noted in the FT-IR spectra (but this can only be surmised at this point). In any event, the infrared (IR) spectra before and after light aging, for both Aquazol 50 and 500, were virtually identical. Following exposure, no additional absorption bands that might be indicative of oxidative processes, or other gross deteriorative processes, were noted in the IR spectra. In particular, the characteristic carbonyl absorption band at approximately 1740 cm^{-1} was not present before or after the light aging; the appearance of such a band after aging might have been taken as indicative of the growth of oxidation products in the polymer.

The pH of solutions of Aquazol 50 and 500 made prior to light aging were identical (6.4 ± 0.1). This value appears to be consistent with the nonionic character of the polymer. The pH of solutions made from aged films of the Aquazol 50 and 500 materials essentially yielded the same value (6.2 ± 0.1). The contribution to the pH from atmospheric CO_2 in both cases was minimized experimentally by making up all solutions in degassed and deionized water.

The DTA data for both Aquazol polymer preparations tested yielded similar plots before and after exposure (Figs. 5 and 6). Under a nitrogen atmosphere, Aquazol 500 exhibited a single endothermic peak at 125 °C; the Aquazol 50 material exhibited a single peak centered at 122 °C. Presumably, the loss of sorbed water and a concomitant crystalline fusion reaction in the polymer in both cases account for these generally observed, single, broad peaks that begin just above 100 °C. It is interesting to note that in both cases the endothermic peaks broaden somewhat on exposure, or resolve themselves into trailing peaks. This broadening is more pronounced with the Aquazol 50 material, which nearly resolved itself into two trailing peaks under the applied temperature gradient. Drops in the molecular weight of the polymers might account for these observed endothermic phenomena. In any event, no exothermic peaks were noted with either of the Aquazol preparations, and there were no signs of decomposition up to the maximum test temperature of 220 °C. The extrapolated T_g's derived from the DTA plots remained at or below the value reported by Chiu and coworkers (55 °C) for samples cast from aqueous solutions of the polymer—which suggests, at least, that no increases in molecular weight were likely to have occurred during exposure. The TGA analysis performed on the Aquazol 500 material after aging confirmed the thermal stability of the polymer (Fig. 4). The data of Chiu and coworkers suggested a stability of the polymer to greater than 400 °C; the light-aged material appeared, if anything, to be more stable against thermal decomposition: 20 °C higher than the value reported for the unaged material.

When standard solutions of unaged and light-aged Aquazol 500 were compared, solution viscosities did appear to drop on light aging (Fig. 8). If the Mark-Houwink equation for the polymer, deduced by Chiu and coworkers, is correct, then the intrinsic viscosity of the exposed samples of both Aquazol 50 and Aquazol 500 appears to decrease, suggesting drops in molecular weight in both cases on aging.

Size exclusion chromatography experiments (Figs. 2 and 3) also tended to indicate that both Aquazol preparations dropped in molecular weight (MW) after light aging. In the case of the Aquazol 50 material, the molecular weight drop was only slight; however, the molecular weight drop of the Aquazol 500 was substantial. The elution position for the Aquazol 50 material, both before and after light aging, was as expected for a polymer with a molecular weight of about 50 K, when compared to standard protein markers run over the same column. The unaged Aquazol 500 material appeared to have an MW closer to 300 K initially; the bulk of it appeared to elute just after the standard marker apoferritin (MW = 443 K).

On light aging, a substantial portion of the Aquazol 500 material appeared to drop in molecular weight to approximately 50 K, and eluted just after the albumin marker (MW = 66 K). With light-aged samples of both Aquazol 50 and Aquazol 500, no insoluble matter (gel fraction) was evident in the material applied to the column heads, and no material was eluted with the breakthrough, or excluded, column fraction. (The nominal exclusion limit for the Sepharose CL-6B used to pack the column is an MW of 1.5 M.)

These results tend to suggest that, under the light-aging conditions imposed, both Aquazol preparations seemed to decrease in size, rather than cross-link or increase in size. In the case of the Aquazol 500, the drop from an initial 300,000 daltons to around 50,000 indicates a chain scission (if that is what is occurring) at a frequency of 1:600 monomer units. This same frequency of chain scission seemed to be occurring in the SEC data from the Aquazol 50 (and is reflected in the only slight drop detected in its molecular weight on aging). It is also interesting to note that, at 218 nm (the wavelength used to detect the eluting polymer), end groups on the polymer may be responsible, in particular, for the increased absorption seen during the SEC experiments. After light aging, the lower molecular weight of the Aquazol 500 gave an overall stronger molar absorption than for the same weight of unaged material on the column.

Re-solubilization tests (Fig. 7) revealed essentially two important features. First, on light aging, both of the Aquazol preparations remained essentially re-soluble in the same solvents in which they were initially soluble. Second, the rates of re-solubility, surprisingly, were not ordered or determined by solvent polarity alone. Water turned out to be a slower (and therefore “poorer”) re-solubilization agent for both Aquazols than acetone, for example, which turned out to be the best (fastest) re-solubilization agent tested. It should be noted that during the tests it was observed that, in general, the slower a particular solvent was at redissolving the Aquazol materials, the wider or broader the range of time-to-separation measurements. Nevertheless, the test method was acceptable because predictable film thicknesses could be generated over and between the glass beads (an inert support) used for the test. More important, the traditional tests for re-solubility, based on the subjective application of solvents with the swab test and hard-to-control variables, could be avoided. The only pressure exerted on the coated test-bead pairs was from the agitation of the test solvent itself by the magnetic stirring apparatus; stirring rates could be easily controlled. Clear end points were always marked by a separation of the two beads that composed the test pair, so there was never a question as to when a certain amount of material had dissolved.

While Chiu and coworkers presented data comparing adhesive properties of Aquazol 500 on various substrates with poly(vinyl pyrrolidone) (PVP) and poly(vinyl alcohol) (PVOH) polymer preparations (the so-called Scotch Tape test), the tensile strength measurements done in association with this study revealed two other important working properties of Aquazol. Since Aquazol is a water-soluble polymer, its moisture absorption—as a function of the ambient relative humidity—could potentially affect its strength as a material. Although films of the polymer were tested at only a few specific RH levels (Table 5), the data indicated that a family of stress/strain curves were generated as a function of RH. Generally, Aquazol exhibits a Young’s modulus characteristic of an extremely plastic material. Yield stresses (s_y) were quite low, varying from

17.85 kg m^{-1} at 73.8% RH to only 44.62 kg m^{-1} at 8% RH. Concomitant elongation to yields (e_y) were small, varying from 50% at 73.8% RH, to 12% at 8% RH. Replicate measures suggested that yield stresses and strains did change with RH, and generally increased slightly with decreasing RH. Nevertheless, the Aquazol films were extremely plastic materials even at low RH levels. Elongation-to-break values (e_B), even at 8% RH, were enormous (380%) at only slightly increased breaking stresses. In comparison with other adhesive materials traditionally used for paint consolidation on wooden substrates, this retention of plastic character, even at low RH levels, makes it a much better choice than, for example, a traditional hide glue which may have an elongation-to-break of only 2–3% at similar low RH values.

Case Studies

In an effort to evaluate the less tangible, but equally important, practical working or handling properties of Aquazol 50 and 500 solutions as consolidants, three painted wooden objects from the Winterthur collection were chosen for testing. These objects had decorative materials that needed consolidation. Only small areas on each object received treatment with the Aquazol preparations; the majority of the surfaces requiring consolidation are to be treated with more conventional materials.

Settee (Winterthur 57.0957). This piece is part of a set with side chairs, originally belonging to Governor and Mrs. Joseph C. Yates of New York. It was constructed sometime between 1800 and 1810, possibly in the Albany area. The piece exhibited pinpoint flake losses over most of the decorative surfaces; generally, these losses closely followed the grain pattern of the wood substrate. The immediate need was for consolidation of the flaking paint and preservation of the design materials. Additionally, however, once the design materials were stabilized, there was a curatorial interest in cleaning the grime-laden paint surface. The goal of the conservation treatment, therefore, was to consolidate the massive amount of flaking paint with a material that would not interfere with or complicate later stages of restoration (i.e., cleaning).

Armchair (Winterthur 57.0973). This armchair is believed to have been constructed in New York City, between 1810 and 1820. Localized areas of paint cleavage were obvious over the surface of the armchair; the losses seemed to be associated more with acute damage to the armchair, along with a history of rough use, rather than an incipient cleavage problem. As with the settee previously mentioned, the immediate conservation need was to consolidate existing design materials. The consolidant would have to be "gap-filling" or sufficiently substantial to secure the loose design materials because of the rather thick, or heavy, off-white paint layer. The curatorial goal at this point was stabilization only; however, cleaning, compensation, and limited inpainting might be attempted at some later point. Whatever the material introduced, it would be difficult to reverse; moreover, it would have to allow for, and be compatible with, future compensating and inpainting materials.

Carved wooden eagle (Winterthur 59.2354). The immediate conservation need for this object—attributed to Wilhelm Schimmel, Carlisle, Pennsylvania, between 1865 to 1890—was consolidation of paint that was poorly adhered to underlying paints; no restoration beyond stabilization was planned. The need for a consolidant that would penetrate effectively between paint layers was paramount. Since no further restoration work would be done on the object, its surface appearance after consolidation was critical to its representation in the collection.

Summary of test consolidation treatments

Solutions of Aquazol 50 were prepared in working concentrations that varied from 10% to 25% (w/v) in water. Solutions were also made in the same weight percentage ranges with water and ethanol blends of up to 10% solvent (v/v). Aqueous solutions of Aquazol 500 were also evaluated at working concentrations of 10–20% of the polymer in water only, and in water-ethanol blends of up to 10% solvent (v/v). Initial consolidation tests were done by applying the polymer solutions by brush, allowing the consolidated areas to dry for a day, and then probing these areas to gauge the effectiveness of the treatment. At this point, no reactivation or heat-sealing properties of the polymers were exploited; only their effectiveness as direct consolidants was evaluated. Initially, a numerical "rating" system was used to evaluate the effectiveness of the various test solutions in each of four specific categories pertaining to working characteristics; ability to flow and penetrate; ability to "relax" flakes; overall security on drying; and visual effect on surrounding design materials.

In summary, it was noted that solutions of Aquazol 50 and 500 in water alone actually penetrated less effectively than aqueous solutions of the polymers that contained small amounts of ethanol. Apparently, the slight decrease in surface tension effected with the added alcohol was useful in allowing for a more complete penetration on the two objects with primarily paint-to-wood adhesion problems (i.e., the settee and the armchair). A 20% solution of Aquazol 500 in a 1:10 ethanol-water blend gave the best results on all three objects. The addition of the alcohol was required for the Schimmel eagle to maximize the penetration into and consolidation of the areas of paint-to-paint cleavage.

The widespread paint cleavage on the settee necessitated that the most friable areas be consolidated by brushing this mixture over both paint and loss areas. After allowing the treated areas to dry, the excess consolidant was successfully removed with water only, even to the point of removing surface grime without any apparent loss of consolidation. The use of a water-soluble consolidant in this way suggests that follow-up aqueous cleaning methods can be used over surfaces consolidated with Aquazol without immediately undoing the consolidant.

In general, it was also noted that more than one application of the Aquazol 50 solutions was required to secure thick paint layers onto the porous wood substrate materials (e.g., the armchair). The initial solutions apparently soaked into the porous wood substrate on application and did not fill the gap between loose paint and the wood; thus, additional applications were required to effect a satisfactory consolidation. The solutions containing Aquazol 500 faired much better in this regard; they generally achieved an effective consolidation with a single application, regardless of the substrate.

Conclusion

The experimental results seem to confirm the observations made by Chiu and coworkers (1986) as to the thermal stability of poly(2-ethyl-2-oxazoline). Viscosity, thermal, and SEC data after light aging suggest that the polymer may tend toward depolymerization rather than cross-linking. The

SEC data seem to be the most telling indicator of this tendency in the present study. From the standpoint of reversibility, or ease in re-solubilization, this tendency may still recommend the polymer as a potential adhesive in conservation. When compared to the aging properties of Acryloid B72 (a standard polymer already used in conservation as a coating or adhesive material) and its tendency to cross-link on aging (Feller 1977), polymers such as Aquazol that tend toward depolymerization may pose less risk in terms of becoming insoluble with age.

However, if the observed chain scission frequency is correct (1 in 600), then preparations of the polymer much greater than 50,000 to 60,000 daltons, used in adhesive situations, may suffer failures concomitant with even this low depolymerization rate. From the tensile strength data, Aquazol appears to be a weak adhesive and may only lend itself to applications where weak forces are at work on adhesive joints. In the present context, the material seemed to perform well as a paint consolidant on wood or paint substrates. Tensile strength data seemed to suggest that, even at low RH levels, the polymer remained very plastic in character. This lack of sensitivity to RH compares favorably with traditional natural consolidants (e.g., hide glue) that may embrittle at low RH levels.

Other characteristics of the polymer that are promising for conservation are the lack of discoloration in the light-aged samples, the water-soluble nature of the resin from a toxicity and safety standpoint, and the absence of a solvent smell.

Certainly, more experimentation should be pursued with this new material. Future directions for research could include evaluating its stability and performance in the context of materials commonly found in painted artifacts. Another avenue of research could (and should) be obtaining and evaluating preparations of the polymer that are methyl tosylate free.

Acknowledgments

The authors gratefully acknowledge the help of Jose Alvarez in the Analytical Department of the Wintherthur Museum; Robert Trent, former curator of furniture at the Winterthur Museum; and Fran Mayhew at the University of Delaware, Textile Science Department.

Notes

1 Chamberlin, T. A. 1977. Madison N.L. U.S. Patent 4 001 160.

2 Aquazol 50 from lot no. 930330; Aquazol 500 from lot no. 930325 (see Materials and Suppliers).

3 Polystyrene chips, 50 mm x 89 mm, from lot no. 1 (see Materials and Suppliers).

4 Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale, PA 15096. Report, issued June 1989, as SAE J1960: Accelerated Exposure of Automotive Exterior Materials Using a Controlled Irradiance Water-Cooled Xenon Arc Apparatus; Chevolet-Pontiac-Canada Group, Headquarters, General Motors Corporation, 30001 Van Dyke Avenue, Warren, MI 48090-9020. Report issued December 1989 as SAE J1885: Polystyrene Reference Plastic Exposure Data.

5 Standard molecular weight compounds were obtained in kit form (see Materials and Suppliers). The column was calibrated with the Blue Dextran (2 mg ml^{-1}, MW 2,000,000), apoferritin (10 mg ml^{-1}, MW 443,000), albumin (10 mg ml^{-1}, MW 66,000), and carbonic anhydrase (3 mg ml^{-1}, MW 29,000) provided in the kit. Aliquot solutions (2 ml) of these compounds in the eluting phosphate buffer were applied individually to the column (with the addition of 5% glycerol as a loading aid, as per Sigma Technical Bulletin no. GF-3), and their elution profiles were tracked.

6 A Perkin Elmer (Lambda 4C CUV-3) scanning spectrophotometer was used.

7 A Corning (model 125 Digital) pH meter equipped with a Corning general purpose combination electrode (model no. 476531) was used to measure the pH of test solutions.

8 Analyses were done on a DuPont 950 thermogravimetric analyzer. The capillary tubes used were DuPont, no. 900302, 2 mm diameter.

9 The glass spheres (which were 5 mm in diameter) were acid washed, rinsed, and air dried prior to use.

10 The forceps were 15.87 cm in length (Fisher Scientific, Inc., cat. no. 13-812-45).

11 A Corning (model PC-353) magnetic stirrer was used in this study, with a 12.7 mm × 7.9 mm octagonal Teflon-coated stir bar (Fisher Scientific, Inc.).

12 An Analect (model RFX-65) FT-IR microscope, equipped with a diamond anvil sample cell, was used (Spectratech model no. 0042-444, Stamford, CT).

13 A Scott CRE-500 tensile strength tester was used for the strength measurements in this experiment (GCA/Precision Scientific Co.).

14 American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

Materials and Suppliers

Aquazol 50 and Aquazol 500, Polymer Chemistry Innovations, Inc., 1691 West College Ave., State College, PA 16801.

glass spheres (cat. no. 11-312C), Fisher Scientific, Inc., 711 Forbes Ave., Pittsburgh PA 15219-4785.

molecular weight compounds kit (cat. no. MW-GF-1000), Sigma Chemical Co., St. Louis MO 63178.

polystyrene chips, Test Fabrics, Inc., P.O. Box 420/200 Blackford Avenue, Middlesex, NJ 08846-0414.

Sepharose CL-6B (cat. no. CL-6B-200), Sigma Chemical Co.

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1978 *The Museum Environment*. London: Butterworths.

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Newman *Tempera and Other Nondrying-Oil Media*
Figures 4, 9: Courtesy of the Museum of Fine Arts, Boston.

Part Two
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Serck-Dewaide *Support and Polychromy of Altarpieces*
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Ravenel *Painted Italian Picture Frames*
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Yorke *Royal Painted Furniture in King Charles I's England*
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Part Three
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Portsteffen *Form and Polychromy*
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Safford *Floral Painting on Early Eighteenth-Century American Furniture*
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Shelton *Short Primer on White-Painted Furnishings*
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Mussey *Verte Antique Decoration on American Furniture*
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Odegaard *Investigation of the Nature of Paint on Wood Objects*
Figures 3 and 4: Courtesy of Arizona State Museum, The University of Arizona.

Harpainter and Augerson *Painted Furniture and Wooden Decorative Arts*
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Part Four
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Marincola and Soultanian *Monochromy, Polychromy, and Authenticity*
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Hulbert *Conservation of the Fourteenth-Century Ceiling at St. Helen's Church*
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Payer et al. *Interior Decor of the Ursuline Chapel*
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Schmidt *Painted Tester Bedstead at Agecroft Hall*
Figures 1–3, 5: Courtesy of the Agecroft Association. Figure 4: Courtesy of Victory Chinnery. Figure 6: Courtesy of Wiltshire County Council Library and Museum Service; and Victory Chinnery.

Gilmore *Hunnewell Cottage*
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Ferrell *Importance of Conservation to Research*
Figures 1–2, 4–6: Photo by Lynton Gardiner. Figure 3: Photo by Carol Warner.

Ray and Reilly *Treatment of Painted Wooden Folk Art*
Figures 1–8: Courtesy of The Colonial Williamsburg Foundation.

Parker and Sixbey *Jeffrey: Horse of a Different Color*
Figures 1–3: Courtesy of Friends of the Carousel Archives.

Part Five
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Samet *Philosophy of Aesthetic Reintegration*
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Hunt *Conservation of Folk Art*
Figures 1–8: Courtesy of the Shelburne Museum, Shelburne, Vermont.

Hastings and Bigelow *Collaborations Past and Present*
Figure 1: Courtesy of Alex Jamison Photography, Arlington, Virginia. Figures 2–3: Courtesy of Hampton National Historic Site.

Gordon *Applied Aesthetics*
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Part Six
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Michalski et al. *The Ultrasonic Mister*
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