RECENT SCIENTIFIC RESEARCH IN PAPER CONSERVATION

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ABSTRACT—Scientific research in paper conservation since 1988 is discussed and summarized. Research on chemical, physical, and optical properties of paper and on the aging of paper is outlined. A synopsis is given of recent findings in paper conservation concerned with washing, bleaching, solvents, enzymes, and sizing.

1. INTRODUCTION

In 1988, the first American Institute for Conservation Book and Paper Specialty Group Update Session reviewed scientific research in five important areas of paper conservation: encapsulation, fumigation, washing, deacidification, and bleaching (Kruth 1988). Research published since then continues to add to the body of knowledge about these and other treatments, and to shed light on the relationship of treatments to paper properties and the aging of paper (Robb 1991). Paper conservators are increasingly concerned about the long-term effects of treatments not only on media, but on the chemical and physical properties of all the components of the paper composite, including the fibers, fibrils, and cellulose polymer chain (fig. 1). This review summarizes scientific research pertinent to these concerns. To provide the context for evaluation of paper conservation research, information regarding paper properties and the aging of paper is outlined first, followed by a synopsis of recent findings about paper conservation treatments involving washing, bleaching, solvents, enzymes, and sizing. The information in this article is highly selective and

condensed, representing only a small portion of the research data. The reader is urged to consult the cited references for more details and additional findings.

2. PAPER PROPERTIES

Burgess and Binnie (1990) advise that "The evaluation of scientific results and recommendations in conservation literature is greatly aided by a good understanding of the rationale behind the selection of test materials and methods." An understanding of the rationale behind research design is enhanced by a knowledge of paper properties and their reaction to relative humidity, temperature, radiation, and pollution. These environmental factors initiate degradation mechanisms of hydrolysis, oxidation, and crosslinking (table 1). These degradation mechanisms can be detected by various tests (table 2), which measure the chemical, physical, and optical properties of paper. The following summary presents a few basic terms and concepts relevant to these properties. (Optical properties are a type of physical property and can be used to monitor chemical change. They are covered separately here for purposes of organization.)

2.1 CHEMICAL PROPERTIES

Chemical properties of a paper sheet are derived primarily from variables of furnish or pulp components and from formation or manufacturing processes (Dwan 1987). Furnish variables include the fiber type; pulp stock;

refining techniques such as beating or bleaching; and composition including fillers, sizes, coatings, and colorants (Priest 1989a). Formation variables include basis weight, calendaring, and manufacture variables such as speed, drainage, drying, and fiber bonding. Each variable in turn has distinct properties. For example, the pulp stock from which a paper is made has specific properties, including crystallinity, fiber bonding, viscosity, and drying, which affect the nature of the final sheet. Refining techniques such as beating affect fibrillation, swelling, hydration, and water retention. On the molecular level, cellulose, the primary and most stable component of paper fibers (fig. 1), has properties imposed by its structure (fig. 2), which creates amorphous and crystalline regions in fibrils (Smith 1991). The amorphous regions are random, flexible, and water accessible (fig. 3), while the crystalline regions are ordered, rigid, inert, and relatively impermeable to water

(Kremer and Tabb 1990). Examples of chemical properties of paper (table 2) and their respective test methods (Waterhouse 1988; Wilson and Parks 1979) are listed below.

- Degree of polymerization (DP) of the cellulose chain, decreased by hydrolysis, oxidation, and chain scission and increased by cross-linking (table 1), can be determined by viscosity measurements or gel permeation chromatography, which also indicates molecular weight distribution.

- Acid content can be measured by alkaline reserve titration, odometric total acid, and surface and cold extraction pH (Burgess and Binnie 1990).

- Production of functional groups such as carboxyl and carbonyl groups, in particular aldehydes and ketones (table 1), through hydrolysis and oxidation, alters the molecular structure. Content can be

TABLE 1

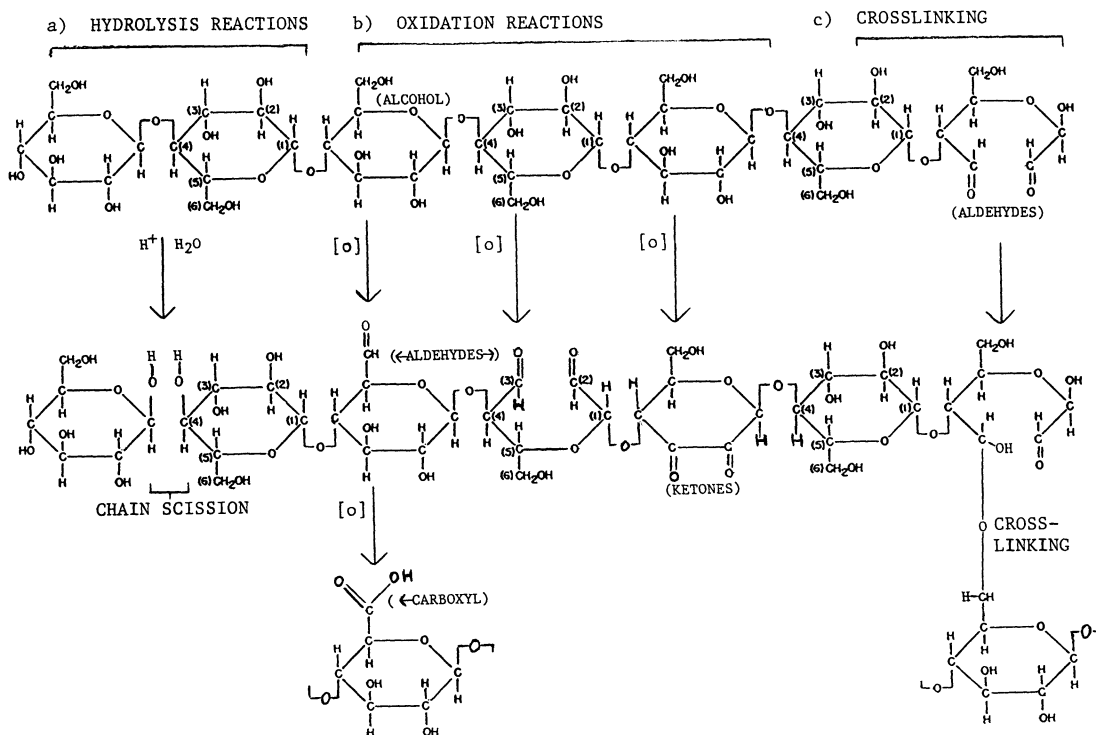SOME ILLUSTRATIVE EXAMPLES OF COMMON CELLULOSE REACTIONS

TABLE 2
TESTS USED TO MEASURE THE EFFECT OF CERTAIN REACTIONS ON SOME
CHEMICAL, PHYSICAL, AND OPTICAL PROPERTIES OF PAPER

Test	Hydrolysis		Oxidation		Crosslinking		Bonding "Order"[1] Decrease		Thermal Decomposition	
	P	S	P	S	P	S	P	S	P	S
2.1 CHEMICAL PROPERTIES										
DP[3]	↓		↓	↓	↑		—	—	↓	
Acid, H⁺		↑	↑		—		—	—	↑	
Carboxyl		↑	↑			↓	—	—		
Aldehyde	↑		↑↓			↓	—	—	↑	
Ketone	—	—	↑			↓	—	—	↑	
Peroxide	—	—	↑		—	—	—	—	↓	
Moisture regain	—	—	↑↓		↓		↑		—	—
Alkali solubility	↑		↑		↓		—	—	↑	
2.2 PHYSICAL PROPERTIES										
Fold	↓		↓		↓			↓	↓	
Tear	↓		↓		↓			↑	↓	
Burst	↓		↓		↓		↓		↓	
Tensile	↓		↓		—	—		↓	↓	
Elongation	↓		↓		↓		↓		↓	
Modulus	↓		↓		↑		↓		↓	
TEA[4]	↓		↓		↓		↓		↓	
Zero span	↓		↓			↓	—	—	↓	
Wet tensile		↓		↑	↑		—	—	—	—
2.3 OPTICAL										
Blue reflectance		↓	↓	↓	—	—	—	—	↓	

Arrows note increase or decrease in measurements indicative of certain degradation mechanisms. For example, a decrease in degree of polymerization (DP) (↓) could indicate the cellulose chain has undergone hydrolytic, oxidative, or thermal degradation mechanisms, but if DP increases (↑), the cellulose may have undergone cross-linking (e.g., as a result of treatment). (Reprinted with permission from Wilson and Parks 1979; Waterhouse 1988) Additional tests for properties, particularly with respect to aging, include fatigue, creep failure, flex tests, ultrasonic, electrical, and newly developed subjective tests (Waterhouse 1988, 60)

Key
Reaction or Change; P = Primary, S = Secondary
1. Includes degradation of bonds formed by sizing agents.
2. Probable occurs only during accelerated aging.
3. Degree of polymerization.
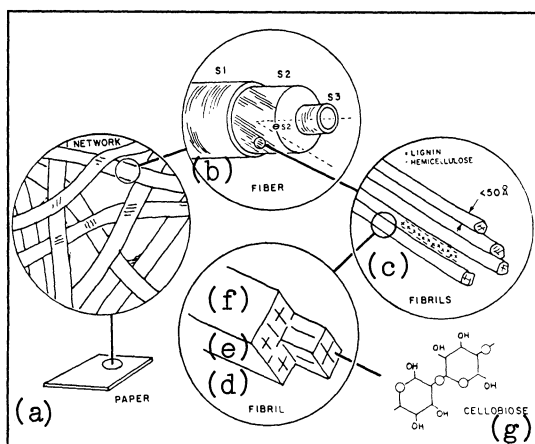4. Tensile energy absorption.

Fig. 1. Paper structure and bonding (two different representations): fig. 1a, paper sheet; fig1b, fiber; fig. 1c, fibril bundle, held by weak van der Waals forces with a bond strength of 2–10; fig. 1d, fibril, composed of microfibrils; fig. 1e, microfibril composed of layers of cellulose chains; fig. 1f, one layer composed of cellulose chains held side-by-side with hydrogen bonds having bond strengths of 3–6 kcal/mole; fig. 1g, cellulose chain composed of glucose units held by strong covalent bonds having bond strengths of c.86 kcal/mole. (Reprinted with permission from Waterhouse n.d.; Dwan 1987; Hollinger 1988)

(g) Atoms of carbon, hydrogen and oxygen combine to form the glucose units which make up the long ribbon-like chain (molecule) of cellulose. This chain or "ribbon" continues for 3000 to 5000 units.

(f) These long ribbon-like chains (molecules) are built up into sheets, which are held together by the side to side hydrogen bonding between the chains. (O...H represents hydrogen bonding.)

(e) The sheets are stacked tightly into layers and held securely together by Van der Waals forces. This is a microfibril.

(d) Microfibrils

(c) Bundles. The microfibrils organize into bundles by the same side-to-side hydrogen bonding and layer-to-layer Van der Waals interaction that formed microfibrils.

(b) Fibres. The fibre is formed from bundles in the same way microfibrils and bundles were formed.
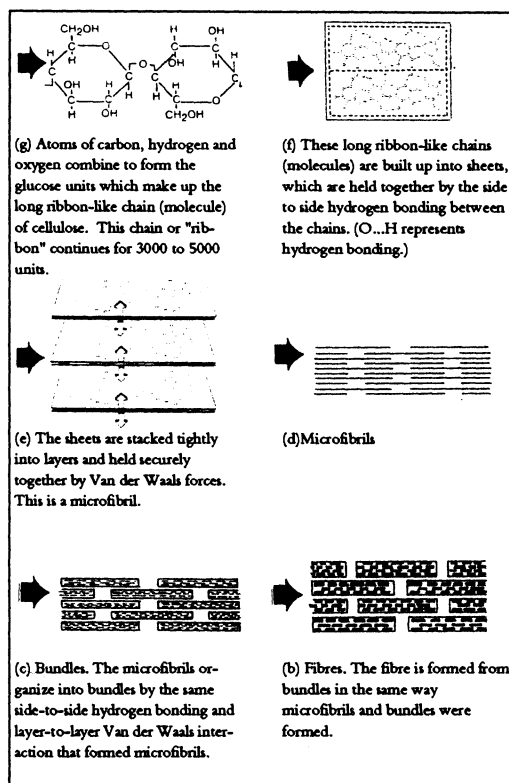
Fig. 2. Detail of fibril, showing crystalline and amorphous regions and proposed aging mechanism (Reprinted with permission from Inaba and Sugisita 1991, after Koura and Krause 1987)
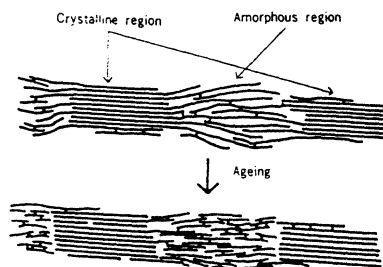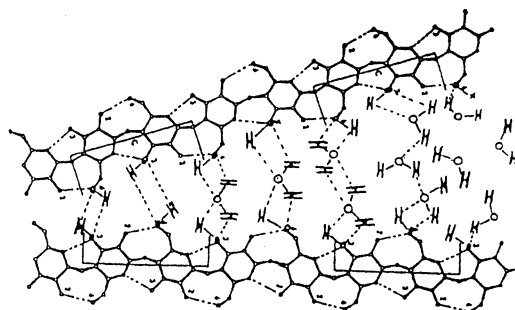

Fig. 3. Detail of cellulosic microfibril amorphous region showing water adsorption (Reprinted with permission from Kremer and Tabb 1990)

measured by several methods including titration and iodometric techniques.

- Peroxide formation, which could occur from oxidation is difficult to measure, but one technique suggested in the conservation literature is measurement by the Russell effect (Daniels 1988).

- Moisture content and regain can be measured by gravimetric methods.

- Alkali solubility includes measurements of carbonyl content by hot–1% alkali solubility test, copper number, and the borohydride reducing method. Ester and ether content can be measured by Raman spectroscopy, nuclear magnetic resonance, and ultrasound.

It is important to note that it is the noncellulosic components in some papers that are the most reactive, as in the case of lignin-containing papers. Ironically, this very instability makes such papers the most difficult to monitor and measure chemically.

2.2 PHYSICAL PROPERTIES

Physical properties of paper sheets are determined by basis weight, thickness, density, two-sidedness, smoothness, permeability, handle, rigidity, roughness, and porosity, which in turn affect sheet properties of softness, hardness, compressibility, dimensional stability, curl, and strength (Casey 1980; Bolam 1962). Attention tends to focus on strength measurements since "strength properties can . . . serve as indications of the permanence of paper, even when the nature of the chemical changes responsible for the deterioration remains unknown . . . [and] can also be used for evaluating the effectiveness of treatments for aging" (Caulfield and Gunderson 1988). The following are some strength, or mechanical, properties and examples of respective test methods.
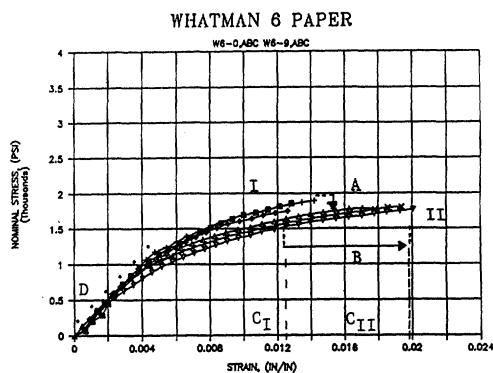

Fig. 4. Example of a stress–strain plot of the machine direction (MD) tensile strength of unwashed (I) and washed (II) Whatman paper (Ca(OH)$_2$:deionized water 1:1), showing A) stress or load to break; B) strain, elongation, or stretch to break; C) tensile energy absorption area (TEA) or total area under the curve, representing work expended to break, and characteristic of durability (e.g., the washed paper [II] may be more durable than the unwashed paper [I] since its total area under the curve [C$_{II}$] encompasses most of and exceeds [C$_I$]); and D) Young's modulus, or initial straight line of the slope characteristic of elastic materials and indicative of stiffness. (Modified from van der Reyden et al. 1988)
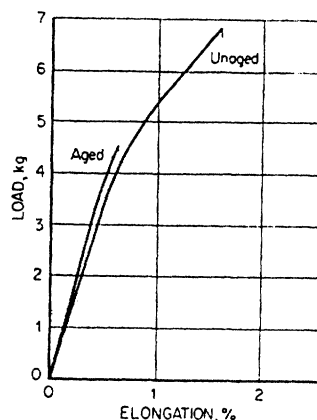

Fig. 5. Load-elongation (stress-strain) curves of paper before and after aging 15 days at 90°C and 50% RH (Reprinted with permission from Waterhouse 1988)

- Fold endurance, hampered by a wide standard deviation owing to the limited area tested, can be measured with the MIT folding endurance tester. This method produces results most consistent with the handling of books.
- Tear resistance, including edge tear resistance or internal tear resistance (similar to the kind of tearing occurring to a nicked book page), can be measured using the Elmendorf tear test.
- Burst strength, one of the most common tests in the paper industry but not much used in paper conservation, is measured by the Mullen test.
- Tensile strength is often determined by standard span tensile strength testing, which measures interfiber bonding and total network strength, and is expressed by stress-strain plots, tensile energy absorption, and Young's modulus (fig. 4). Fiber strength, internal bonding, or fiber orientation may be measured by zero-span, z-direction (through-the-thickness), and wet tensile tests.

2.3 OPTICAL PROPERTIES

As mentioned, optical properties are physical properties which conservators often monitor as an indication of chemical changes. Optical properties of paper include color, brightness, whiteness, reflectance, gloss, opacity, transparency or transmittance of light, and fluorescence (Mark 1983). Measurements are made with reflectometers, spectrophotometers, colorimeters, and goniometers. Findings may be reported in various modes including percent reflectance (figs. 6a, 8a), CieL*a*b* (fig. 9e) and Kubelka-Munk scattering and absorption coefficients (Berndt 1989; Burgess and Binnie 1991).

Paper properties are interdependent. Interpretation of test data is complicated by the fact that measurements represent "a combination of such factors as flexibility, bonding strength, and fiber strength . . . which are dependent on type of fibers, length and thickness of fibers, imperfections in fibers, flexibility, pattern of fiber network, number of bonds, strength of the individual bonds, the weight of the paper, the apparent density, the moisture content, and many other factors" (Casey 1980). Properties are in turn affected individually and collectively by environmental parameters such as temperature, humidity, and light; by aging; and by conservation treatments.

3. AGING OF PAPER

The primary concerns of research on the aging of paper involve determination of 1) the effects of natural aging on paper and 2) the parameters of accelerated aging that most nearly match the effect of natural aging on paper.

3.1 NATURAL AGING

Natural aging may cause paper to become discolored and cellulose to become more crystalline and stiff, as has been shown for accelerated aging (fig. 5). Crystallinity can be measured by x-ray diffraction, acid hydrolysis, or moisture content, although the percent crystalloids depends on the method used and on interference of additives (Daniels 1986). Koura and Krause (1987) concluded that, based on changes in mechanical properties, treatments using ammonia could reverse the degree of crystallinity in aged papers. Discoloration of a paper sheet may be caused by the formation of chromophores upon aging as a result of exposure to, among other things, light (Lee et al. n.d.) and volatile gases (Daniels 1988). Daniels has hypothesized that oxidation can be induced in paper by hydrogen peroxides from oxidation of lignin, leading to autoxidation of paper (Daniels 1988). He has described how

hydrogen peroxide can be recorded by the Russell effect, which produces an image on sensitive photographic film. More recent studies have focused on the effects of pollution on aging of papers, particularly deacidified papers, which appear to have a greater capacity to absorb sulfur dioxide and nitrogen dioxide (*G.C.I. Newsletter* 1990).

3.2 ACCELERATED AGING

Accelerated aging, undertaken at elevated temperatures and humidities, may initiate reactions resulting from thermal degradation. These reactions could, depending on conditions and length of exposure, produce products different from those of natural aging (Feller 1987). Erhardt has used gas chromatography mass spectrometry to analyze extracts of papers aged at different temperatures and relative humidities. He found that humid oven aging of Whatman #1 filter paper (at 90°C and 100% RH) hydrolyzed cellulose and xylan, producing mostly glucose and xylose, also found in naturally aged papers. Dry oven aging at 90°C and above produced very little glucose. Aging at 150°C produced a different set of products (Erhardt et al. 1987; Erhardt 1988). Other researchers have focused on the effects of cycling aging conditions, finding that cycling relative humidity increases the degradation of single sheets of paper (Shahani et al. 1989). However, the effects of cycling appear to be less pronounced for books, the format of which may slow down changes. On the other hand, simulated book materials tend to age faster in comparable accelerated aging conditions than do single sheets, perhaps owing to the trapped concentration of degraded materials forming what amounts to an adverse microclimate.

As noted by Erhardt (1988), there are many standards for accelerated aging applied to paper. For instance, TAPPI test methods lists three dif-

ferent sets of conditions ranging from 90–105°C and 0–100% RH depending on the type of papers and property to be measured, while ISO requires 80°C and 65% RH. In general, a wide variety of aging conditions continue to be used in current research, including the studies described in the following section.

4. CONSERVATION TREATMENTS

The investigations reported in this section address changes to paper properties caused by various treatments. Conservators are cautioned to remember that the demands of good research limit the scope of experimental design, which cannot possibly be expected to incorporate all conditions of actual conservation treatment practice. The practicing conservator should recognize that reported data pertain to specific experimental conditions. However, the data may be correlated to actual practice by extrapolation based on an understanding of the interaction of paper properties. For instance, one might hypothesize that a bleach found to damage cellulose in a cotton paper may also damage cellulose in a paper made of more reactive ground wood pulp. By the same token, changes to properties caused by one solvent application technique may not occur with an alternate application technique. The conservator must also recognize that the interpretation of research data requires great care. Treatments cause changes to more than one property. Some property changes may be perceived as beneficial while others may be considered detrimental, and indeed, these initial assessments may be reversed over time as the object ages. Each treatment of an object must be considered individually to decide whether the net benefits outweigh the disadvantages, depending on the needs and character of that particular object. For example, washing might remove discoloration, but this benefit must be balanced against a

loss or change in sizing. Sizing agents such as gelatin impart strength to paper (Barrett 1989). A loss of sizing might reduce breaking length by decreasing tensile strength. At the same time, a loss of sizing might improve fold endurance by decreasing stiffness. The changes in properties may or may not be relevant, depending on subsequent conservation treatment (i.e., resizing) or the needs of the object. For instance, an improvement in fold endurance might be more beneficial for paper used in a book than in flat artwork. Furthermore, experimental conditions must be carefully considered when comparing data from different projects. Minor variations, for instance in techniques of sample preparation involving wetting or drying, may inadvertantly affect properties of gloss or tensile strength. Modifications in degree of solvent saturation and direction of application, or in drying time, pressure, and technique, must be taken into account. All of these variables are factors that a conservator must consider when formulating treatment options, particularly regarding the most intrusive conservation treatments of all: those involving washing or deacidification, bleaching, solvents, enzymes, and sizing.

4.1 WASHING

Washing in paper conservation refers to overall treatment of paper with aqueous solutions, usually containing additives (such as wetting agents, solvents, or alkaline compounds) that serve to swell paper fibers and dislodge or dissolve impurities in order to aid in cleaning, neutralize acidity, or deposit an alkaline reserve to guard against future acid exposure (Daniels and Shashoua 1991). Early research found that washing with deionized or distilled water could cause a drop in pH and fold endurance, since such highly purified water can dissolve the stabilizing magnesium and calcium components of paper (Tang and Jones 1979; Nelson et al.

1982). On the other hand, alkalinization, either with low concentrations used for cleaning or higher concentrations used to deacidify paper, may cause color change, especially in papers containing lignin, the unstable impurity in ground or mechanical wood pulp (Burgess et al. 1990). In order to differentiate papers that may be improved with selected washing techniques from those that may not, Burgess has analyzed naturally aged papers that were washed in deionized water and in water containing low (20 ppm) and high (200 ppm) concentrations of magnesium compounds, before and after humid thermal accelerated aging. Washing with deionized water did remove some beneficial calcium and magnesium salts. However, since washing also removed acid impurities, sample papers with high initial acidity that were washed actually maintained after aging a degree of polymerization greater than the unwashed untreated controls, which underwent more acid hydrolysis during aging. On the other hand, the degree of polymerization for papers with low initial acidity did indeed drop, relative to the controls, after washing with deionized water followed by accelerated aging. In addition, although some alkaline-treated, lignin-containing papers maintained greater mechanical strength with zero-span tensile measurements, an increase in concentration of some alkaline magnesium components produced an increase in yellowing of the lignin-containing papers. As a result, the authors concluded that, pending further studies on lignin-containing papers, institutions should "consider reducing the quantity of ground wood papers which are being treated by aqueous deacidification," shifting priorities to materials with little or no lignin (Burgess et al. 1990).

Increase in yellowing may also occur in ligneous ground or mechanical wood pulp treated by solvent immersion (Lienardy and van
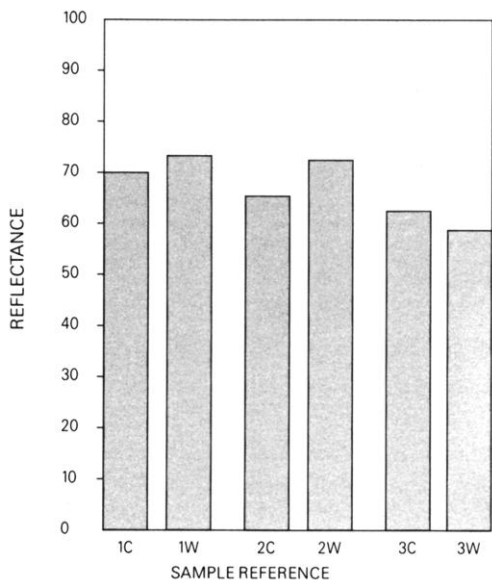
Fig. 6a. Whiteness (reflectance) of rag paper (1), chemical woodpulp paper (2) and chemical/mechanical woodpulp (3); Control sample (C), Washed sample (W).
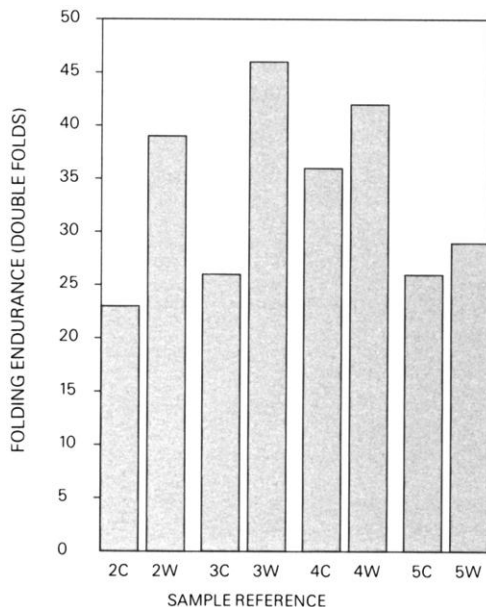

Fig. 6c. Fold endurance of chemical woodpulp paper (2) and mechanical woodpulp papers (3–5); Control sample (C), Washed sample (W).
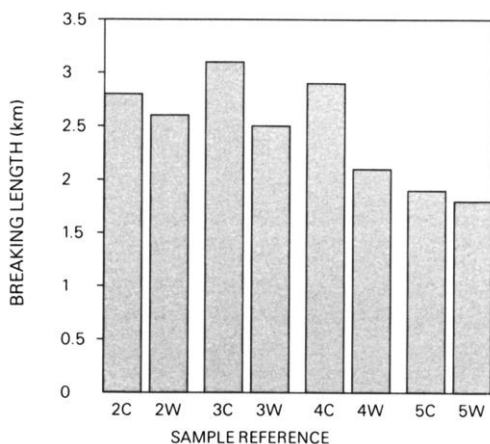

Fig. 6b. Tensile strength expressed as breaking length: chemical woodpulp (2) and mechanical woodpulp papers (3–5); Control sample (C), Washed sample (W).

Fig. 6. Effect of solvent washing on paper: fig.6a, reflectance; fig.6b, breaking length; fig.6c, folding endurance   (Reprinted with permission from Lienardy and van Damme 1990.)

Damme 1990). Ligneous paper washed with 30% isopropanol in tap water registered a drop in reflectance as compared to the control (fig. 6a). For all wood pulp paper samples, immersion for 5 minutes in alcohol (followed by washing in water for 30 minutes), caused an increase in fold endurance but a reduction in breaking length (figs. 6b and 6c). Breaking length is the calculated upper limit of length of a uniform paper strip that would support its own weight if it were suspended at one end. It is a convenient measure for comparing the tensile breaking strength of papers of varying grammages (Caulfield and Gunderson 1988). A similar effect of alcohol was found in an earlier study, in which immersion of a mixed cottonwood pulp, alum rosin sized paper in 9:1 ethanol:deionized water appeared to cause a slight reduction in elongation to break (figs. 7a
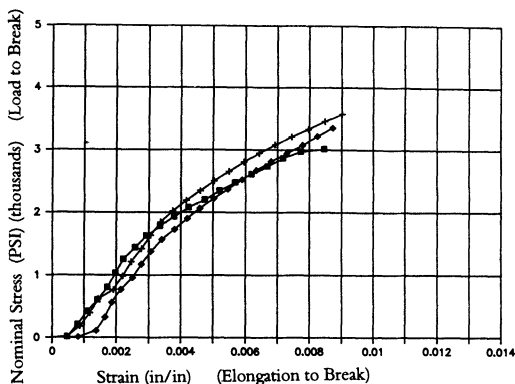
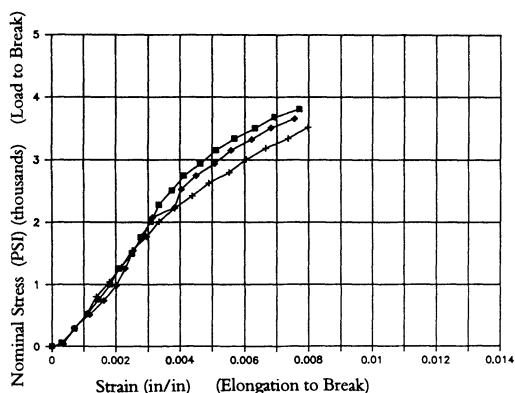Fig. 7a. Aqueous light bleached, 24 hours. Naturally aged Strathmore paper.

Fig. 7b. Ethanol light bleached, 24 hours. Naturally aged Strathmore Paper.

Fig. 7. Comparison of elongation to break (MD) in samples treated in water (fig. 7a) and ethanol:water 9:1 (fig.7b). (Reprinted with permission from from van der Reyden et al. 1988)

and 7b) as compared to samples immersed in water alone (van der Reyden et al. 1988).

Reductions in breaking length, or in elongation to break, could result from a breakdown of sizing or the disruption of hydrogen bonding in paper immersed in organic solvent, which may in effect dehydrate or debond the paper. On the other hand, immersion in water swells fibers and releases the strain built into paper during the original drying of the sheet and subsequent

aging. Aging stiffens paper as the random array of cellulose chains in the flexible amorphous regions of fibrils become more ordered and crystalline, held rigid by hydrogen bonds between the cellulose chains (fig. 2). Washing introduces water molecules that sever these bonds in the amorphous regions (fig. 3). Water can act as a plasticizer, swelling fibers and allowing fibrillar rearrangements that build in creep and encourage rebonding at less stressed sites upon drying. This phenomenon may account in part for the increase in stretch or elongation to break after washing (unsized) paper in alkaline solutions (fig. 4). Stretch is regarded by some in the paper industry as "one of the most important criteria for satisfactory behavior of paper in use" (Higgins and de Yong 1962). Furthermore, "a stress-strain curve in which the initial slope (Young's modulus) is high, where there is little curvature, and where the value for elongation to break is low, indicates a brittle material. A tough material is one that exhibits more viscoelastic behavior, a pronounced curvature, and a high elongation to break" (Caulfield and Gunderson 1988).

4.2 BLEACHING

Bleaching, potentially damaging to paper and fibers, is nonetheless occasionally used by some paper conservators when washing fails to remove stains. Many paper conservators have become particularly interested in aqueous light bleaching, which consists of bleaching paper immersed in an alkaline solution by exposing it to light (Keyes 1980). Feller investigated the role of pH in aqueous light bleaching and found little difference in bleaching efficiency among solutions with pH 7–10, either before or after aging, on thermally discolored filter paper (fig. 8a) with little lignin or hemicellulose content (Lee et al. n.d.). He also found insignificant photochemical damage as manifested by color

reversion or change in degree of polymerization after accelerated thermal aging of aqueously light bleached filter paper samples (fig. 8b). This parallels findings regarding mechanical properties of an earlier study (van der Reyden et al. 1988), which has been expanded to include accelerated aging (Schaeffer et al. n.d.). The tensile strength of cotton-fibered Whatman #1 paper aqueously light-bleached with UV filtration in an Atlas Weather-Ometer is statistically identical to all washed controls, with an increase in elongation remaining greater after aging than the aged untreated control (figs. 9a, 9b). On the other hand, a naturally aged artists' paper with similar fiber composition but heavily sized with alum and gelatin underwent a reduction in stress to failure or load to break for samples immersed in a solution of calcium hydroxide (pH 9.5) in the Weather-Ometer (figs. 9c, 9d). This situation is probably the result of a breakdown in sizing, especially when the paper is subjected to thermal degradation during aqueous light bleaching in the (warm) Weather-Ometer. The reduction in strain, or elongation to break, before aging might be attributable to thermal or photooxidation of the paper or sizing. However, after aging the strain measurements of both types of immersed samples are statistically identical to each other. Interestingly, the aqueously light-bleached samples underwent the least color reversion of all samples, particularly in yellowing, as measured by CieL*a*b* (fig. 9e). Lienardy and van Damme (1990) reported similar results for samples aqueously bleached by unfiltered sun and artificial light, although the samples underwent a drop in fold endurance, which may indicate ultraviolet degradation and/or a breakdown or alteration in the sizing of their naturally aged test papers.

Naturally aged test papers pose several problems with respect to good research design
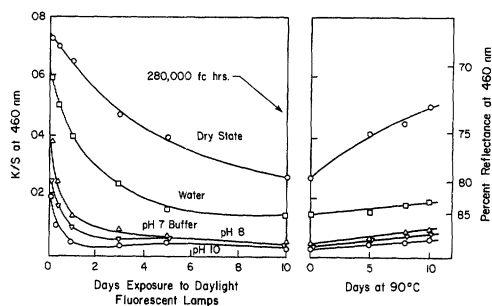

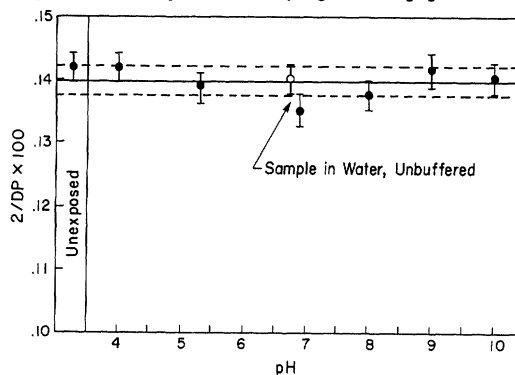Fig 8a. Bleaching of thermally-aged filter paper

Fig. 8b. Relationship between DP and pH of solution during exposure of aged filter paper to DFL for six days

Fig. 8. Effect of pH on bleaching efficiency (fig. 8a) and degree of polymerization (fig.8b) (Reprinted with permission from Lee et al. n.d.)

since such papers vary considerably in fiber, size, additives, and degradation products even within a single sheet. Such papers may also have low degrees of polymerization and tensile strength, which make changes difficult to measure. Consequently, naturally aged papers should be tested along with controls of similar fiber composition, which can be thermally pre-aged, and may provide the outer parameters for treatment evaluation. If relatively strong papers (such as the filter and artists' papers made by Whatman) are damaged by a treatment, naturally aged papers
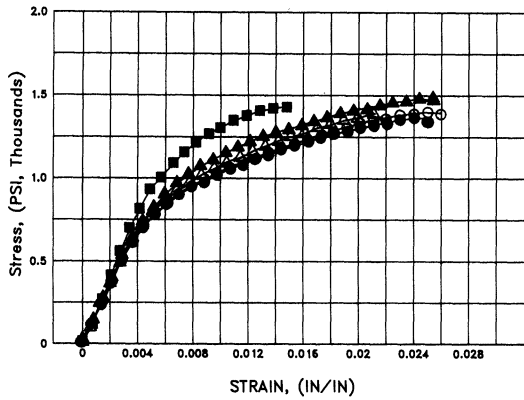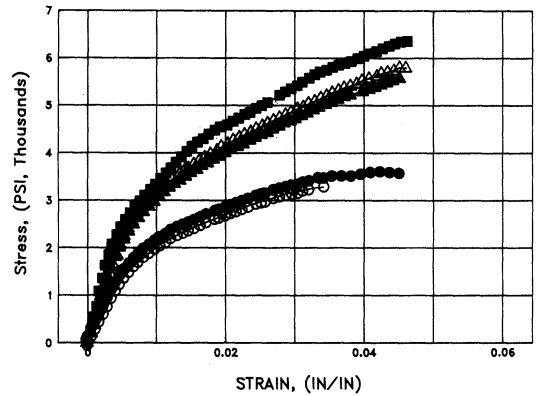
Figure 9a. Whatman 1, Unaged. 2 hours in Weather-Ometer

Figure 9b. Whatman 1, Aged. 2 hours in Weather-Ometer
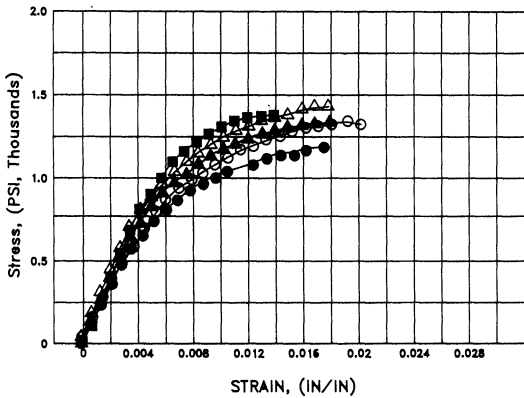

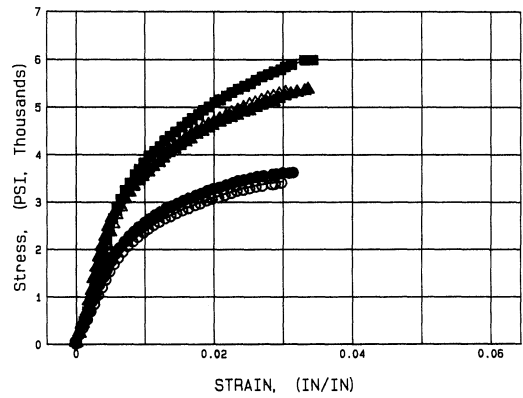Figure 9c. Whatman 56, Unaged. 2 hours in Weather-Ometer

Figure 9d. Whatman 56, Aged. 2 hours in Weather-Ometer

Fig. 9. Effect of aqueous light bleaching on tensile properties (MD) of unsized and sized paper, before and after aging. Figs. 9a, 9b. Treated unsized paper underwent an increased in elongation as compared to unwashed control both before and after aging. Figs. 9c, 9d. Sized, naturally aged artists' paper underwent a decrease in load to break in washed samples as compared to control as a result of breakdown in sizing, and particularly in aqueously light-bleached samples, which had the highest solution temperature (40°C+), which may have thermally degraded sizing even further. Key: Controls: ■Untreated, dry, in dark; ▲ Washed Ca(OH)2, dry, in dark; △ Washed Ca(OH)2, dry in light; ● Washed Ca(OH)2, Immersed Ca(OH)2, in dark. Experimental Sample: O Washed Ca(OH)2, Immersed Ca(OH)2, in light. (Schaeffer et al. n.d.)
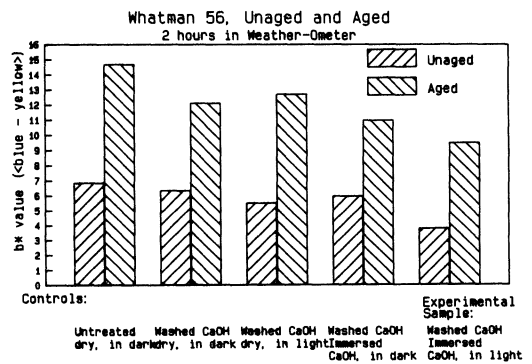

Figure 9e. Whatman 56, Unaged and Aged. 2 hours in Weather-Ometer. Effect of aqueous light bleaching on color stability of a gelatin-sized paper before and after aging: b* values of CieL*a*b* (increase in b* indicates increased yellowing)

of lesser quality may be expected to be damaged as well.

The effects of aqueous light bleaching have been compared to those of chemical bleaches (van der Reyden et al. 1988; Lienardy and van Damme 1989; Hofmann et al. 1990; Flieder et al. 1991). Findings before and after aging indicate that aqueous light bleaching, with UV filtration, causes no significant damage to the mechanical strength of a preaged cotton paper as compared to chemical bleaches (fig. 10) when all bleaching conditions are followed by extensive washing (Hofmann et al. 1992). Regarding chemical bleaches in general, Burgess (1990a) has shown that although both hydrogen peroxide and sodium borohydride produce excellent color stability with respect to reversion, increasing the pH of stabilized hydrogen peroxide to pH10 caused a decrease in the degree of polymerization by 21% in a 19th-century rag paper. However, sodium borohydride can actually stabilize damaged cellulose, reducing the color-producing double bonds of two types of carbonyls (aldehydes and ketones) back to original colorless alcohol species (Burgess 1988).

4.3 SOLVENTS

Use of solvents can often preclude the need to bleach, by removing stains and adhesive residues, but paper conservators remain concerned about the effects of solvents on paper (MacKay and Smith n.d.). One problem, for instance, is that local treatments can sometimes produce tidelines which are only visible in ultraviolet light, or after aging of the treated object. In addition to the publications mentioned in the washing section, recent studies have focused on particularly solvent sensitive papers such as modern chemical watermarked papers (Watkins 1990), historic transparent paper (Flieder et al. 1988; Richardin et al.
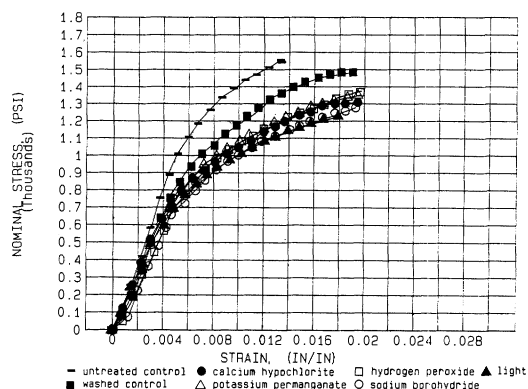
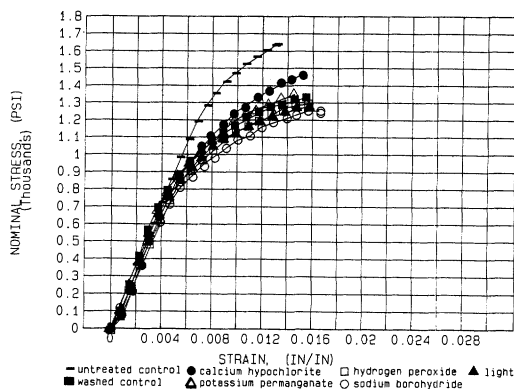Fig. 10a. Whatman 1 (pre-aged): Before accelerated aging

Fig. 10b. Whatman 1 (pre-aged): After accelerated aging

Fig. 10. Comparison of aqueous light bleaching to some traditional chemical bleaches with respect to tensile properties (MD), both before (fig. 10a) and after (fig. 10b) aging. The sample paper was thermally pre-aged to simulate natural aging before treatment. (Source: Hofmann et al. 1992)

1990), and coated papers (Baker et al. 1989). Such papers are affected by both choice of solvent agents and choice of application technique. Recent work has shown that choice of solvent diluent (such as ethanol, acetone, or toluene), affects properties of selected polymers (Hansen et al. 1991). A study comparing solvents and application techniques for stain removal (using immersion, suction table, and poultice) has documented alteration of surface finish of paper, including break-up of impregnants, leading to a

loss of translucency in tracing papers (fig. 11).
The effects of solvents on coated papers parallel
in some ways findings by paintings conservators
on the reaction of pigment films to solvent
swabbing and immersion (Hedley et al. 1990).
For example, application of aqueous poultices to
some coated papers may produce what appear
to be tideline stains but are actually cracks (fig.
12). Attempts to reduce stains on coated papers
with solvents could actually increase cracking
(van der Reyden n.d.). Clearly, there is a need
to continue to characterize specialty papers
using Fourier transform infrared spectroscopy,
gas chromatography/mass spectrography, and
scanning electron microscopy/energy dispersive
spectrometry in order to track effects of alterna-
tive solvent application techniques with SEM
dot mapping and UV reflectance/transmission
microscopy.

4.4 ENZYMES

Like many other conservators, paper conser-
vators employ enzymes to break down adhesives
among other things.   Burgess (1990b) has
reported on criteria for enzyme selection and on
the discrepancies between the working proper-
ties and information found in specifications on
manufacturers' labels.   Labels may record en-
zyme activity measured on substrates such as
casein (which is different from adhesives
generally encountered by paper conservators), as
well as the pH at which the enzyme was as-
sayed, rather than the pH for optimum activity
(Grattan et al. 1987).   In fact, some enzymes are
biphasic, having high activity levels in both acid
and basic pH ranges (fig. 13). Although enzyme
activity approximately doubles in rate for each
10°C rise from 20–60°C, activity can be in-
creased at lower temperatures by increasing the
concentration of enzyme.   However, another
study, in which amylase enzymes were tagged
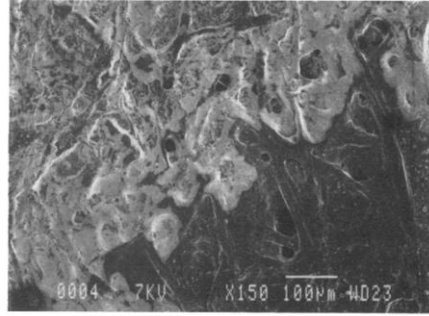with radioactive iodine isotopes, indicated that

Fig. 11a . Treated in the upper left section
by immersion in ethanol, which caused a
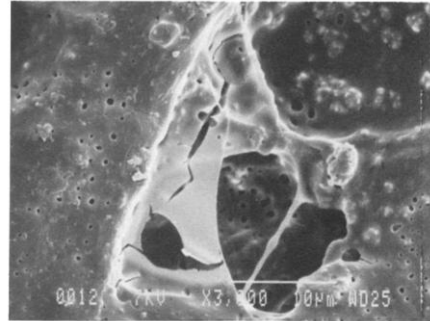breakdown in the impregnant [150 x]
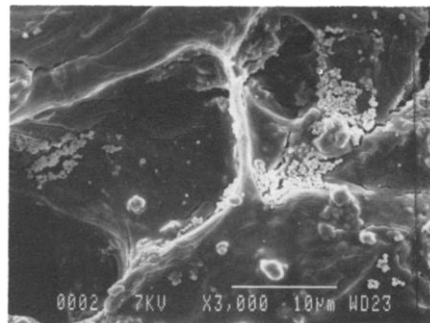

Fig. 11b. Detail, untreated area [3000x]

Fig. 11c. Detail, treated area [3000x]

Fig. 11. Scanning electron micrographs of a
tracing paper (transparentized by impregna-
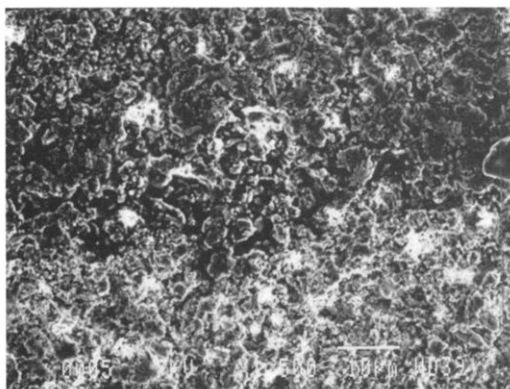tion with an acrylic resin)

Fig. 12. Scanning electron micrograph of a prepared ground paper (coated with barium sulfate and calcium carbonate in acrylic resin) treated with an aqueous poultice, which caused cracking along the perimeter of the poultice
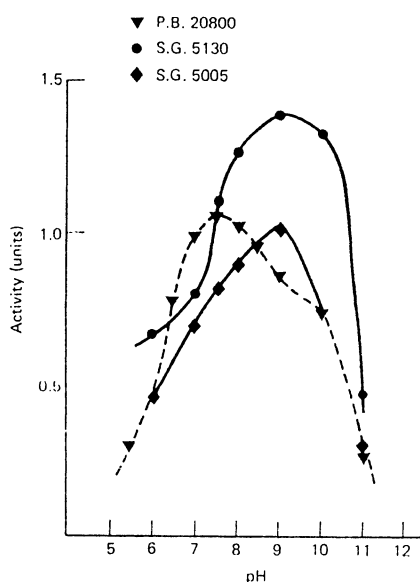

Fig. 13. One example of biphasic enzyme activity of amylase P.B. 20800 with increased activity around pH 7 and 10. (Reprinted with permission from Grattan et al. 1987)

even though most of the enzymes were removed by rinsing the test papers twice with water, the amount of enzyme residue remaining in the paper after rinsing, although quite small, increased with an increase in concentration and purity (Andrews et al. 1990). The authors therefore recommended that conservators use the minimum amount of enzyme needed to do the job.

4.5 SIZING

Paper conservators are concerned about whether any of the solutions discussed above alter or remove the sizing in paper, whether papers should be resized, and if so, with what (Barrett 1989). Barger has examined papers, from 17 disbound books, which after washing and deacidification were resized with gelatin, parchment, wheat starch, methylcellulose, and Klucel-G (Carapella et al. 1990). SEM examination revealed that the surface relief of the papers selected for treatment played a large role in the conformation and adhesion of sizing. For instance, gelatin adhered more poorly to high-surface-relief rag papers than to more planar wood pulp paper. The sizes tested did not appear to undergo significant morphological change after accelerated aging, aside from some curling and cracking of gelatin size. However, Feller's study of cellulose ethers found poor color stability, after accelerated aging, for hydroxypropyl cellulose powders like Klucel-G, with a 28% decrease in reflectance (Feller and Wilt 1990). Hydroxypropyl cellulose powders appear to be more susceptible to thermal degradation than other cellulose ethers because of a 60% degree of substitution of hydroxyls by etherifying reagents. Feller's study also found that in general, cellulose ethers with lower molecular weights tend to discolor more than those with higher molecular weights.

5. CONCLUSION

Evaluating conservation research is especially challenging because of the diversity in research design, test conditions and procedures, and the interpretation of results and recommendations. The importance of good research design—requiring a limited area of investigation, reduction of variables, thorough characterization of samples, and familiarization with the chemistry of materials in order to understand the advantages and disadvantages of selected test procedures—cannot be overemphasized (Burgess and Binnie 1990). On the other hand, procedures are continually being developed and refined to improve instrument sensitivity and the quality of information, and this may account for apparent inconsistencies in research. For instance, there is a need to codify accelerated aging conditions such as temperature, relative humidity, time, sample mounting techniques, and air-to-sample volume ratios, to determine the best modes for color measurements (e.g., the use of whiteness, brightness, reflectance, or CieL*a*b* or Yxy modes, relative to standard or target values, and recorded as difference or absolute measurement); and to further improve and adapt chemical and mechanical tests. Consequently, because of the constant evolution of procedures, it is imperative that all data be recorded and available for reinterpretation. Conservators must evaluate conclusions and recommendations in the context of the specifics of a particular research project, and conservators alone bear the responsibility for determining how findings apply to their own special cases. Researchers can supply only increments of information, which often generate more questions, and research findings can only aid, not replace, a conservator's good judgment.

tions of aluminum sulfate in gelatin sized papers. Finally, I would like to thank the many interns and colleagues who participated in projects mentioned above or reviewed and critiqued the manuscript, including Konstanze Bachman, Katherine Eirk, Eleanor McMillan, Nancy McRaney, Alan Postlethwaite, Olga Souza, Chris Tahk, Charles Tumosa, and Lambertus van Zelst.

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