MODERN TRANSPARENT PAPERS: MATERIALS, DEGRADATION, AND THE EFFECTS OF SOME CONSERVATION TREATMENTS

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ABSTRACT

Modern transparent papers, also known as tracing papers, require specific chemical and physical properties, such as translucency, smoothness, and whiteness, which are produced by specially formulated compositions and manufacturing procedures. Ironically, these special formulations may in some ways render such papers susceptible to problems from damage and degradation, while also making them particularly reactive to moisture and solvents that must be used in conservation treatments to correct these problems. In order to evaluate the effects of water and organic solvents on such papers, four research projects were designed to consider the variables of paper composition, properties, type of solvent, technique of application of solvent, and flattening. This paper summarizes findings on materials characterization, degradative effects of aging, and some effects of solvents used on transparent papers for stain reduction, humidification, and flattening.

INTRODUCTION

Most museums, libraries, and archives contain large numbers of a class of material generically known as transparent or tracing papers, which serve, among other things, as the support layer for technical and architectural drawings. Such drawings are used frequently by architects, engineers, historians and other scholars for many research purposes, and are often exhibited and reproduced. However, they can not be effectively replaced by photographs, facsimiles, or microfilm, since the design media may be difficult to decipher as it is often faded, smeared or smudged from handling. In addition, the drawings may have notations, added or erased, which indicate significant changes in designs important for an understanding of the history or evolution of architectural ideas and concepts, and these subtle but important changes may not be captured in reproductions, which also fail to accurately portray the variety of media, representing different elements and stages of design. Finally, reproductions occasionally crop writing scribbled in margins or on the reverse of the drawings. Consequently, scholars often require access to the original material. Unfortunately, the composition, size, age, past use, and fragility of these drawings, compounded by their frequent handling, makes them particularly vulnerable to damage. To prevent the total destruction of the original information, such drawings frequently require conservation treatment.

Conservation treatment of such artifacts is complicated not only by diverse and sensitive media, but also by of the very nature of the paper supports, which require specific chemical and physical properties, such as translucency, whiteness, and smoothness, produced by specially formulated compositions and manufacturing procedures. However, these special formulations and procedures may render such papers exceptionally susceptible to damage such as surface marring, embedded grime and stains, and degradation such as embrittlement and discoloration. Often the papers are extremely hygroscopic, which renders them particularly susceptible to cockling, planar distortions, and dimensional changes. Different manufacturing processes may also render the papers extremely sensitive to conservation treatments. This is especially true for treatments requiring solvents for washing or deacidification [16, 18, 48], lining with aqueous, solvent-activated or thermoplastic adhesives [3, 4, 9, 19, 21, 36, 39, 40, 48], consolidation [48], stain-removal [5, 17], or humidification and flattening [15]. The papers may also be sensitive to treatment application techniques, such as immersion or use of a suction disk or table [15, 22, 25, 42, 48].

RESEARCH DESIGN

Some conservation literature exists on the analysis and treatment evaluation of old transparent papers [15, 16, 29, 32], but less is available on modern transparent papers [21, 35, 39]. In 1987, the Conservation Analytical Laboratory began research characterizing several types of specialty papers, including coated and transparent papers [5], to enable conservators to 1) categorize the nature of many types of specialty papers in order to anticipate potential changes to properties from aging and solvent treatment, and 2) design treatments selecting solvents and application techniques appropriate to specific types of specialty papers. The ongoing work includes the four interrelated projects described below.

RESEARCH DESIGN FOR MODERN TRANSPARENT PAPERS PROJECT

					· · · · · · · · · · · · · · · · · · ·				
		PROJECT I. CH	ARACTERIZATION (OF EXPERIMENTAL SAMP	LES				
SELECTED SAMPLES FOR:	FIBER PROCESSING:	OVERBEATING:		NATURAL TRACING PAPER SAMPLE "G"					
	SHEET PROCESSING:	ACID IMMERSION:		GENUINE VEGETABLE PARCHMENT PAPER SAMPLE					
		CALENDERING:		IMITATION PARCHMENT PAPER SAMPLE					
		COATING AND/OR IMPREGNATING:		VELLUM PAPER SAMPLE "C"					
	!			PREPARED TRACING PAPER SAMPLE "A"					
IDENTIFI- CATION OF MATERIALS BY:	GENERAL OBSERVATIONS								
	MANUFACTURER'S INFORMATION								
	ANALYSIS:	SEM IMAGING		SEM/EDS	FIIR	GC/MS			
MEASUREMENT OF PROPERTIES:	APPEARANCE:	COLOR		OPACITY	GLOSS				
	PHYSICAL:	STRENGTH		DIMENSIONAL STABILITY					
	CHEMICAL:	РН							
PROJECT II. EFFECTS OF ACCELERATED AGING									
PROJEC	CT III. EFFECTS OF S	OLVENTS & APPLIC	PROJECT IV. EFFECTS OF HUMIDIFICATION & FLATTENING						
WATER	ETHANOL	ACETONE	TOLUENE	IMMERSION	HUMIDIFICATION CHAMBER	HUMIDIFICATION PACK			
	APPLICATION	TECHNIQUES:	FLATTENING TECHNIQUES:						
IMMERSION	POULTICE	SUCTION DISK		AIR DRY	BLOTTER PRESS	SUCTION TABLE			

Project I: Characterization: Samples of modern transparent papers were selected representing four primary categories (natural tracing papers, genuine vegetable parchment paper, imitation parchment paper or glassine, and vellum or prepared tracing papers) (Fig. 1). Characterization included identification of furnish material by fiber microscopy and staining, SEM/EDS, FTIR, and GC/MS, and of formation procedures by SEM imaging and UV microscopy. The samples were also characterized by measurement of chemical and physical properties including pH, color, gloss, opacity, transmission, mechanical strength, and dimensional stability.

<u>Project II</u>: Aging: Evaluation of the effect of accelerated aging on the properties of the selected transparent papers was done by measuring properties, before and after aging, of color, opacity, transmission, gloss, and tensile strength (Figs. 2, 3, & 4). Accelerated aged samples were also used to simulate aged or old papers for treatment and further testing.

<u>Project III</u>: Solvent application: Evaluation of the effects of four commonly used stain-removal solvents applied by three different treatment techniques to transparent papers has included measurement of opacity, gloss, and color (Fig. 5), and subjective observation of appearance (tidelines or ringing) in visible and ultraviolet light, and tracking dislocation of furnish materials by SEM and UV microscopy (Fig. 6) [42, 43].

<u>Project IV</u>: Humidification: Evaluation of the effects of three humidification techniques and two flattening techniques used for transparent papers included measurement of changes in dimensions, transmission and gloss (Figs. 7 & 8), tensile strength (Fig. 9), and SEM surface imaging (Fig. 10) [22]. Conditions of testing and analysis are listed in Appendix 1.

PROJECT I: CHARACTERIZATION

General terms used to describe transparent paper in the literature include tracing paper, oiled paper, onion skin paper, and waxed paper [1, 3, 4, 11, 35, 37, 47]. US Federal Specifications for Tracing Papers UU-P-561H 1972 currently recognizes four classes, with specific minimal requirements for fiber type (100% rag or chemical wood pulp), permanence, translucency, whiteness, and strength, and for properties of tear resistance, fold endurance and opacity, among other things [14, Appendix 2]. Properties may vary depending on intended use. For instance, surface finish might be rough for pencil work or smooth for ink illustrations. Manufacturers of modern transparent papers tend to categorize their products as a) natural tracing papers, b) vegetable parchment paper (genuine parchment paper or pergamet), c) imitation parchment (pergamyn or glassine), or d) vellum paper or prepared tracing papers. Each of these papers is made by either 1) processing the fiber stock by overbeating to insure that the fiber structure is broken down to eliminate air/fiber interfaces, and/or 2) processing the paper sheet to insure that the air pockets are eliminated or filled with a material having a refractive index similar to the paper fibers. A paper sheet can be made transparent by adding or applying a transparentizer (coating and/or impregnating agents), by immersion of the sheet in acid, and/or by calendering. Fiber processing is the primary procedure used to make a) natural tracing paper (highly overbeaten), while sheet processing is used to produce b) vegetable parchment paper or pergamet (immersed in acid), c) imitation parchment, pergamyn or glassine paper (highly calendered), or d) vellum or prepared tracing paper (both made with transparentizers).

Several papers were selected randomly to represent the aforementioned types of modern transparent papers. Glassine paper serves as a "transparent" control, since it has the fewest additives. The compositions of some of the papers are listed and described below; morphological structures have been documented by SEM imaging of surfaces and cross-sections [42, 43].

Fig. 1: SUMMARY OF GENERAL CHARACTERIZATION OF SELECTED MODERN TRACING PAPERS

		NATURAL SAMPLE "G"	VEGETABLE PARCHMENT	IMITATION PARCHMENT	VELLUM PAPER SAMPLE "C"	PREPARED TRACING SAMPLE "A"
M A N U F A C T U R E I N F O	ADDITIVES	sulphamic acid; artificial clay; rust inhibitors; defoaming agents; saponified oils; talc; nitrogen polymer or acrylonitrile/ butylacrylate copolymer	unfilled	unfilled	melamine formaldehyde, urea formaldehyde	silica, aromatic solvent
		surface size: styrene maleic anhydride compound, or quaternalized polymer; modified starch	unsized	unsized	internal and external size: starch	coating: styrene ester, cellulose ester
A N A L Y S I	SEM IMAGING	fibers visible on surface but not in cross- section	fibers visible on surface & cross section	fibers pressed & visible in cross sect.	coated surface; impreg. cross.	heavily coated surf.; impreg. cross.
	SEM/EDS	S, Si, Ca, Hg traces: Al, Cl, Na	Si, Ca, Al, S	Al, Si traces: S, Cl, Na, Ca	Al, S; traces: Si, Cl, Na, K, Ca	Al, Si, Cl traces: S, Na, K, Ca
	FTIR	melamine (?) acrylate, oil	NA	NA	melamine, starch, syn. res.	styrene acrylate, alcohol polymer
	GC/MS	oil	NA	NA	oil	oil, resin

A highly overbeaten natural tracing paper sample, which we have designated "G", is made from a furnish of chemical wood pulp and numerous additives. It has the greatest degree of fibrillation of chemical wood pulp on the surface and virtually no fiber structure in cross-section. This is because the paper is produced in a large volume of water having only 6% fiber content while at a high temperature (80 degrees C), in order to soften the fibers and increase fibrillation. It is machine calendered. The density ranges between 1.1-1.3 grams/cubic cm.

The genuine vegetable parchment paper is made of chemical wood pulp and transparentized by a momentary immersion in sulfuric acid, which changes the wood fibers into an amyloid gel. The gel is then solidified by washing and neutralization, which bonds the fibers into a solvent resistant paper with high initial wet strength recommended by its manufacturer for off-set lithography and silk-screen printing. Testing has not been completed for this paper, so no findings

are included in the current publication.

The calendared imitation parchment paper sample, or glassine control sample, shows in both surface and cross-section SEM photomicrographs highly fibrillated chemical wood pulp fibers, partially "gelatinized" by prolonged beating of the pulp, and highly compressed by calendaring. Glassine chemical woodpulp fibers are beaten in water with a 20-30% fiber content. The final sheet is dampened and then supercalendered, under high pressure and heat (approximately 2000+lbs/linear inch at 180-200 degrees C).

A vellum paper sample, "C", is made from 100% cotton fibers which do not fibrillate upon overbeating as well as chemical wood pulp. The twisted shape of cotton fibers prevents close conformation and traps air, leading to scattering of light at the fiber/air interface. Therefore, such paper requires transparentizers, made up of a coating and/or impregnant. This sample has an

internal and external size of starch and melamine formaldehyde resin.

A prepared tracing paper sample, "A", is made of 100% "rag", and is technically in the same category as sample "C". However, it appears to be coated with a viscous material pitted by minute air pockets, as seen in SEM surface images. In SEM cross-section, this coating appears to impregnate the fibers, which are none-the-less distinct and appear less fibrillated than the other papers. It is transparentized by an aromatic solvent based synthetic resin and top coated by styrene and cellulose esters.

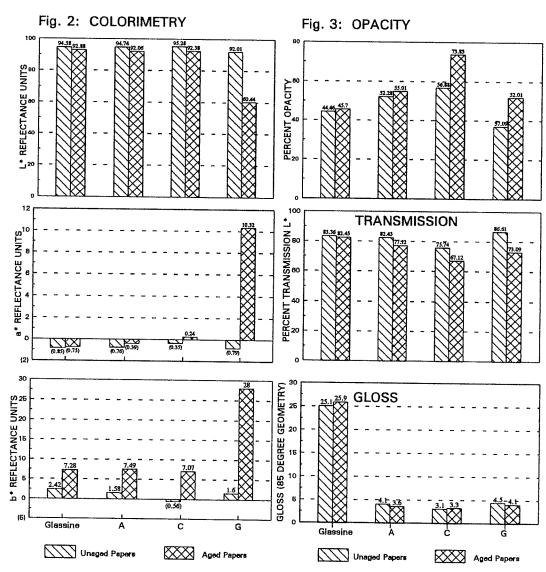
PROJECT II: DEGRADATION FROM AGING

The transparent papers in this study were artificially aged at 90 degrees C, 50%RH for four weeks which, it must be noted, substantially exceeds the conditions required by the Federal Specifications for Tracing Papers UU-P-561H 1972 (14, App. 2). To facilitate comparison with samples characteristic of more standard papermaking processes, measurements for untransparentized Whatman papers have been included in some graphs.

Colorimetry (Fig. 2): The transparent papers for the most part appeared to be about as stable as the glassine paper control, showing a slight increase in yellowing (b*). The notable exception was sample "G", which showed considerable change in all values upon aging, darkening with a shift to red and yellow, and becoming a uniform brown color. This could be due to the presence of chromophores formed, as a result of our severe aging conditions, from the numerous additives in this particular paper. The manufacturer attributes the color change under our aging conditions to the presence of sulphamic acid (amido sulphuric acid) that is added to control sizing, flocculation and algae growth during processing, or to a modified starch added to surface sizing.

Opacity, Transmission, and Gloss (Fig. 3): Aging resulted in an increase in opacity that was visually apparent for all samples, although samples "G" and "C" underwent the greatest measurable increase. All the samples had a corresponding drop in percent transmission. Glassine was the only sample having high initial gloss, owing to supercalendering, and accelerated aging caused little change.

Strength (Fig. 4): The tensile strength of two tracing papers were measured and both had far greater strength than normally processed paper. This may reflect a combination of exceptionally strong sizes, impregnants and/or overbeating. Sample "G", which underwent pronounced overbeating to increase fibrillation and bonding, had substantially greater strength (ultimate stress) and stiffness, exceeding that of prepared tracing paper "A" (transparentized by coating and impregnation), or a heavily sized paper (W56). After accelerated aging, the papers tested underwent a reduction in strength, characteristic of embrittlement. This may be a result of a



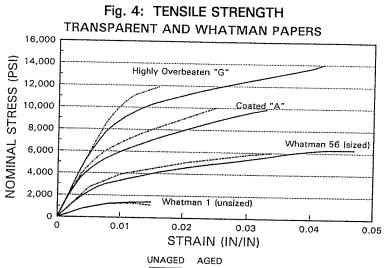
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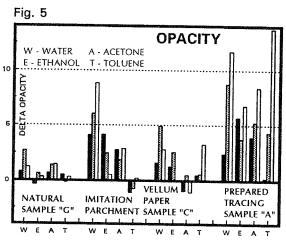


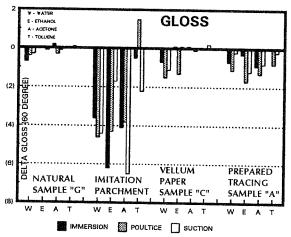
breakdown in sizing. In addition, overbeating, as in the case particularly of sample "G", may be responsible for substantial decreases in strength (ultimate stress) and elongation (ultimate strain), because it produces shorter fibers and weakens fibrillar attachment to fibers.

<u>pH</u>: Glassine and sample "A" had the highest initial surface pHs (6.05 and 5.8 respectively) which dropped slightly after aging (5.4 and 5.1). Sample "C" had a pH (4.3) which increased after aging (5.6). Sample "G" had a low initial pH (4.65) which dropped appreciably after aging (3.8).

PROJECT III: EFFECTS OF SOLVENT TREATMENTS ON TRANSPARENT PAPERS

Transparent papers, because of the special properties mentioned above, tend to be particularly susceptible to treatments with water or organic solvents. For the purpose of this project, four common stain removal solvents (water, ethanol, acetone, and toluene) were applied to samples of each transparent paper by three common conservation techniques (immersion, suction disk and poultice). Evaluation of changes in opacity, gloss, and planar uniformity caused by solvent type, application technique, and paper type indicates certain trends summarized below.





Solvent Evaluation: Water generally caused the greatest net changes of all solvents regardless of application technique or type of paper in this study, resulting in an increase in opacity and decrease in gloss (Fig. 5). Water was the only solvent that caused appreciable planar distortions, which were about the same regardless of application technique or type of paper. Acetone, ethanol and toluene generally caused less effect on opacity and gloss.

Application Technique Evaluation: Suction application frequently caused the greatest increase in opacity and poultice the greatest in gloss. Visual changes induced by poultice may result in part from residual poultice material. Suction application appears to have the greatest effect on cross-section morphology as seen with SEM imaging.

Paper Type: The slightly overbeaten and highly calendared glassine sample underwent the greatest loss of gloss following solvent treatment. The natural tracing sample "G" was the least affected by all treatment conditions. The heavily coated prepared tracing sample "A" had the greatest general reactivity to all solvents tested. Only immersion in toluene had no readily apparent affect on "A", whereas suction disk application of toluene caused an increase in opacity readily apparent in visible light. However, ultraviolet

illumination revealed that areas treated by toluene applied by immersion and poulticing underwent reduced fluorescence and increased absorption, indicative of a break up of surface coating. As already suggested, this paper may be prepared with a toluene based resin. SEM imaging of this paper confirmed that each solvent affected the paper differently [41, 42, 43]. For instance, acetone applied by suction disk appeared to cause a loss of material (Fig. 6a). Immersion of the sample in ethanol appeared to cause a breakdown of material (Fig. 6b), precipitating small spheres which were identified by SEM/EDS as silicon based (Fig. 6c), concentrated in the areas of treatment as evidenced by SEM elemental dot mapping (Fig. 6d) of the same immersed area (Fig. 6b).

Fig. 6a Acetone applied by suction disk (right side of SEM photomicrograph)

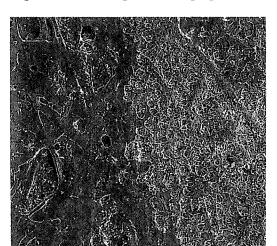


Fig. 6b Ethanol applied by immersion (the upper left)

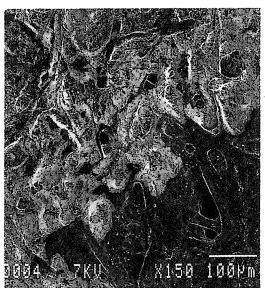


Fig. 6c Detail of 6b identified as silicon

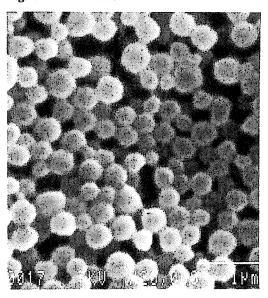
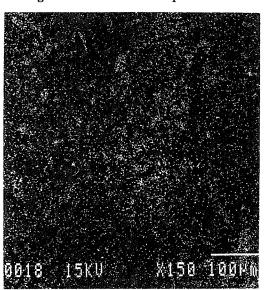


Fig. 6d Elemental dot map for Si in 6b



PROJECT IV: EFFECTS OF HUMIDIFICATION AND FLATTENING ON TRANSPARENT PAPERS

The fourth project investigated the effects of three humidification techniques (using immersion, an ultrasonic humidification chamber, and a humidification pack system) and two flattening techniques (on a suction table and in a traditional blotter press with c. 1PSI weight). Each technique was applied to the unaged transparent papers (to simulate the effect on "new" paper) and to aged paper (to simulate the effect on "old" paper). Evaluation of transmission, gloss, dimensional changes, and, in some cases, strength, indicate certain trends depending on humidification and flattening technique and paper type, as outlined in the following summary (Fig. 7 and Fig. 8).

Fig. 7 DELTA PROPERTIES AFTER HUMIDIFICATION AND FLATTENING OF UNAGED TRANSPARENT PAPERS ("NEW")

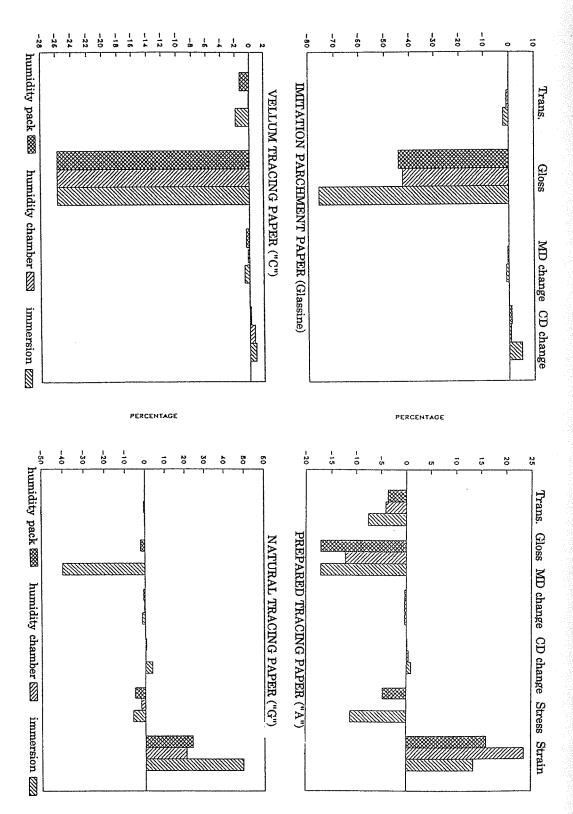


Fig. 8 DELTA PROPERTIES AFTER HUMIDIFICATION AND FLATTENING OF AGED TRANSPARENT PAPERS ("OLD")

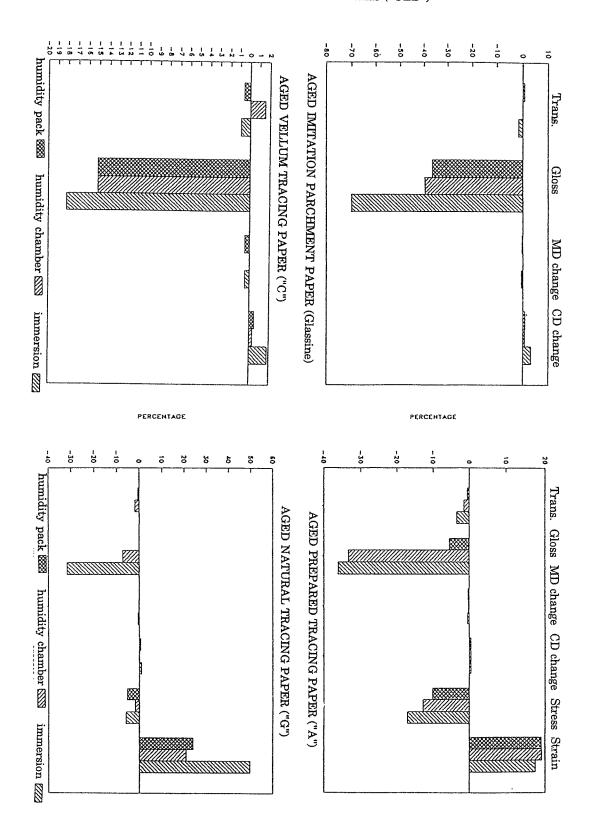
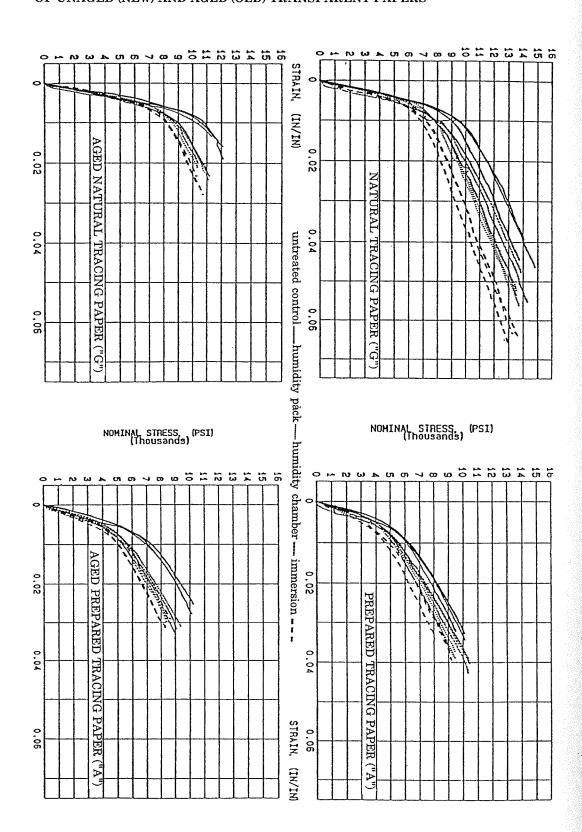


Fig. 9 TENSILE STRENGTH AFTER HUMIDIFICATION AND SUCTION TABLE DRYING OF UNAGED (NEW) AND AGED (OLD) TRANSPARENT PAPERS



Water Application Technique Evaluation: The most severe planar distortion occurred with immersion, particularly for the overbeaten natural tracing "G", followed by humidification pack and chamber, which caused less planar distortion since both techniques effectively reduce water penetration. Immersion also caused the greatest dimensional changes (Figs. 7 & 8), and the greatest change in mechanical properties (Fig. 9), especially in comparison to humidity chamber. This was particularly true for the overbeaten natural tracing paper "G", for which immersion caused the greatest increase in strain to break, possibly from rebonding after the release during immersion of dried-in strain produced during manufacture. Immersion caused a greater decrease in the stress and strain to break for prepared tracing paper sample "A", possibly from the break up of the coating material.

<u>Flattening Technique Evaluation</u>: With respect to the two flattening techniques, compared to air dried controls, suction table drying of immersed samples caused severe distortion, especially for the overbeaten natural tracing "G", as compared with drying in a traditional blotter press. Dimensional changes in the cross-grain direction were greatest for the overbeaten and calendared papers, following immersion and drying on the suction table (Figs. 7 & 8). The coated papers were less affected dimensionally.

<u>Paper Type</u>: Heavily coated prepared tracing sample "A" incurred the greatest change in opacity following immersion, becoming mottled. This may result from a disruption of material which occurred to some degree for this paper regardless of humidification technique. As evidenced by SEM photomicrographs, immersion (Fig. 10a) and a humidification pack system (Fig. 10b) appeared to result in actual loss of the surface material in some areas.

Fig. 10a After immersion humidification

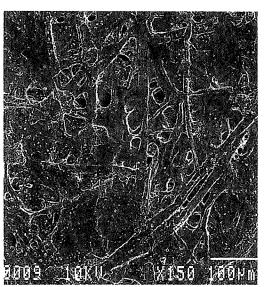
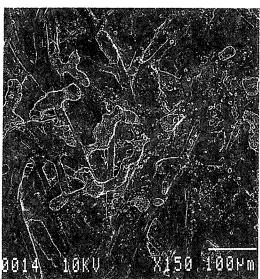


Fig. 10b After humidity pack



CONCLUSION

Some preliminary observations, which may aid conservators faced with the treatment of transparent papers, may be summarized for each project as follows:

<u>Project I</u>: Based on manufacturers' information, literature, and our own findings, there is a great deal of variation and overlap in the furnish and formation procedures for modern transparent papers, and this can make them difficult to distinguish. However, the furnish, formation and properties of modern transparent papers follow certain trends sufficiently different that conservators may be able to determine, with qualifications, whether a paper is characteristic of prepared tracing or vellum paper, a natural tracing paper, a vegetable parchment paper, or

imitation parchment. This may be done, with varying degrees of accuracy, by several techniques, for instance: a) observing general appearance (high gloss for calendared imitation parchment, high translucency for natural tracing paper, high fluorescence for papers with transparentizers), planar and dimensional stability (greater for coated tracings), reactivity to water (greater for overbeaten natural or imitation parchment papers) or organic solvents (greater for heavily coated tracings), and strength (weaker for overbeaten natural tracings after aging); b) analyzing fiber content with polarized light microscopy (chemical wood pulp for natural tracings and parchment papers, cotton for tracings with transparentizers) or sheet morphology by SEM; and c) undertaking instrumental elemental analysis using SEM/EDS, FTIR, or GC/MS. In addition to the more frequently used GC/MS, microtome cross-sectioning of samples combined with SEM, FTIR, and UV microscopy appears to have great potential for providing detailed information on composition and distribution of furnish materials in samples.

<u>Project II</u>: Accelerated aging under the conditions outlined in this paper caused dramatic differences in the properties of all the samples, sometimes in excess of the standards considered acceptable by the US Federal Specifications [14, App. 2]. There are substantial differences in mechanical properties, before and after aging, depending on whether the samples were transparentized by overbeating or by transparentizing agents. In general, the heavily coated transparent paper sample seemed to have better retention of optical and strength properties after aging than the overbeaten natural tracing paper and imitation parchment paper samples.

<u>Project III</u>: The effects of solvents on the surface of transparent papers vary a great deal and seem to depend first on the composition of the paper, followed by the application technique and type of solvent. Of the papers studied here, the natural tracing paper was the least affected and the heavily coated paper was the most affected by the various solvents and application techniques. Water effected the greatest changes (increase in planar distortion and opacity and decrease in gloss) and toluene the least. The property most severely affected was translucency, which decreased in most cases. Prepared papers were the most sensitive to tidelines.

<u>Project IV</u>: Different techniques for humidifying and flattening transparent paper affect properties in different ways. Based on our findings, conservators who must immerse a transparent paper in water might find dimensional changes less severe if the paper is dried in a blotter press rather than on a suction table. Transparent papers with heavy coatings that might soften on exposure to moisture should be humidified in a humidity chamber rather than by prolonged contact with a humidification pack system. This latter technique, however, might be more appropriate for uncoated or natural transparent papers, since initial curling is prevented.

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APPENDICES

- 1. Conditions of testing and analysis
- 2. Summary of US Federal Specifications for tracing papers (UU-P-561H 1972)
- 3. Bibliography

pH measurements: The pH was measured with a Corning Model 12 Research pH meter with an Orion model No. 81-35 flat surface combination electrode. The rinsed electrode, with a pendent drop of deionized water, was lowered onto a square paper sample(1.5 x 1.5 cm) on a polyethylene bag padded with blotters. The pH was recorded after 5 min. The electrode was calibrated with pH 7 and pH 4 buffer solutions before each measurement session. The surface pH of the untreated new and aged controls of each paper type was measured (i.e. 1 measurement for each of 8 samples).

<u>Microscopy</u>: Microscopy was undertaken on a polarized light stage microscope, using transmitted and reflected visible and ultraviolet illumination. UV illumination employed a short pass (Kurz pass) KP 500 excitation filter and a chromatic beam splitter (TK510/K515).

<u>Microchemical staining:</u> For the iodine potassium iodide test for starch, 0.13 g iodine were dissolved in a solution of 2.6 g potassium iodide in 5 ml water. The solution was diluted to 100 ml (Browning 1977, 91).

<u>SEM:</u> SEM imaging and SEM analysis were carried out on a Jeol JXA - 840 A scanning electron microscope with Tracor Northern TN 5502 energy dispersive x-ray analysis system. For imaging the samples were mounted on aluminum stubs and gold coated. For elemental analysis the samples were mounted on carbon stubs and carbon coated.

<u>FTIR</u>: FTIR analysis was carried out on a Mattson Cygnus 100 Fourier Transform Infrared Spectrophotometer with a Spectratech IR-Plan Microscope. The surfaces of the bulk paper samples were analyzed by reflectance; for transmission spectra, fibers were pressed in a diamond anvil cell. To isolate coatings, samples of the papers were extracted with solvents; the solvents were evaporated and the residues analyzed by transmission in the diamond cell.

Gas chromatography: Samples of new papers C, A and G and of the aged paper G were hydrolysed in potassium hydroxide (10% in methanol) overnight, neutralized with 3M hydrochloric acid, extracted with diethyl ether, and then taken to dryness in a stream of nitrogen. The sample was redissolved in methylene chloride and then an equal volume of dimethyl formamide-dimethyl acetal was added to form methyl esters. The prepared sample was analyzed on a Carlo-Erba model 5360 gas chromatograph with a 30 m x 0.32 mm DB-1 column. One microliter of the sample plus a comparable volume of methylene chloride was injected. The injector temperature was 300°C. The initial column temperature of 50°C was immediately raised 10°C/min to 320°C. The effluent was detected with a flame ionization detector at 325°C. The chromatograms of the samples and that of a standard solution of methyl esters ("K101") were compared.

Artificial aging: The transparent paper samples were aged for four weeks in the dark at 90°C and 50% relative humidity in an Associated Environmental Systems HK-4116 Temperature/Humidity chamber. These conditions have been chosen as suitable for artificial aging studies [13]. A set of transparent papers was sewn with cotton thread into plexiglas frames so that all four corners were anchored and the samples did not touch one another. However the samples did vibrate in the oven draft.

Colorimetry, Transmission and Opacity: Color (specular reflectance included), total transmission (diffuse plus regular transmission), and opacity were measured with the HunterLab Ultrascan Spectrocolorimeter (D₆₅; 10° observer, diameter of area of view 1.2 in) using the CIE L*a*b* color notation, where L* represents the degree of brightness (100 white, 0 black), a* the degree of redness (positive numbers) or the degree of greenness (negative numbers) and b* the degree of yellowness (positive numbers) or the degree of blueness (negative numbers). Due to irregularities of the transparent papers, the standard deviation for the L* value of transmission was rather high, sometimes amounting to 0.5. For color and transmission five measurements were taken per sample and averaged. Opacity for the solvent treatment was measured by the ratio of reflection when samples were read against a white and black tile on the HunterLab Ultrascan Spectrocolorimeter.

Gloss: The gloss was measured with a Dr. Lange Labor-Reflektometer RL. Three measurements per sample were taken and averaged at the each of the following angles: 20°, 60° and 85°.

Tensile tests: The tensile properties were investigated using the Mecklenburg relaxation tensometer [28] with a horizontal load applied in the machine direction to the paper strips. Narrow strips of uniform width were cut with a mat cutter. After measurement of the paper thickness in five places with a micrometer, the paper strips were mounted horizontally in the apparatus exposed to laboratory atmosphere. After an initial equilibration period at a gauge length of 2.5 in, the strip was stretched 0.0025 inch and one minute later the stress sustained by the paper strip was recorded. This process was repeated once per minute until the paper strip broke. Measurements were made on three strips of each paper. From these data, nominal stress (force applied per cross-sectional area of the strip) and strain(change in length divided by gauge length) were computed. Nominal stress was plotted as a function of strain for each paper strip. The new and aged untreated controls and the new and aged treated samples of paper A and paper G that were dried on the suction table were measured three times each.

<u>Dimensional changes:</u> Dimensions of the samples were measured once per sample in mm (+/- mm) in cross and in machine direction before treatment, after humidification, after one day drying, after two weeks drying and after two weeks storage following drying. The dimensions of all 56 samples including new and aged untreated controls and new and aged treated samples were measured.

<u>Planar distortion:</u> New and aged untreated controls and new and aged treated samples were compared and subjectively ranked in daylight, raking light and transmitted light in a preliminary attempt to evaluate planar distortion. A total of 56 samples was ranked (ranging from 1 for least changed to 5 for most changed). A blind study is planned to provide more statistically accurate data.

Moisture: Moisture content was measured with a Sovereign electronic moisture meter, model 452 A. The samples humidified with Gore-tex were taken out of the sandwich and placed between polyester film supported by a blotter. The upper polyester film was removed and the surface of the paper measured. The same procedure was applied for the humidity chamber samples. The samples dried out very fast. The measurements had to be taken very quickly. The immersed samples were taken out of the bath and placed on polyester film supported by a blotter. One set of new (120 x 170 mm) and old (90 x 150 mm) untreated papers was cut for this purpose for each humidification technique. Four measurements were taken per paper sample and averaged. A total of 12 samples was measured.

<u>Poultice</u>: Diatomaceous earth (hydrated silica from diatom plant skeletons) was selected for its working properties since, unlike gel poultices (methylcellulose, agarose, starch paste, or hydroxypropylethylcellulose) it can be mixed with aqueous or non-aqueous solvents to form a plaster or paste that absorbs solutes as it dries to a powder, which can then be brushed off. It is more cohesive than fused silica. It is whiter than Fuller's earth, which is formed from hydrated silicates of magnesium, calcium, aluminum, or other metals. It is more controllable than organic solid poultices such as powdered cellulose, paper, or cotton. The diatomaceous earth was saturated with each solvent (approximately 1-2 ml solvent to 0.3 grams earth depending on solvent) and placed on the sample. Contrary to normal practice, the wet poultice was not surrounded by dry poultice which would reduce tideline formation.

<u>Suction disk:</u> Solvents were applied locally by dropper (6 drops of solvent, with drying between drops) on a 15 cm fritted glass bead disk (masked off with polyester film), which can reach a pressure of c. 25"Hg [45].

<u>Suction table</u>: The transparent papers were dried under low vacuum on a Nascor dual mode suction table, which can reach a pressure of c. 4.5"Hg [45].

<u>Humidification pack system:</u> The humidification pack system consisted of a damp blotter placed on a polytetrafluoroethylene membrane (a polyester felt laminate, 1/16" thick, produced by W.L. Gore & Associates, Inc.) which was held in contact with the transparent paper while under light weight (c. 1PSI).

APPENDIX 2: Federal Specifications

The US government has developed specific minimal standards for modern tracing papers. According to Federal Specifications (UU-P-561H 1972), modern tracing papers are classified as four types which are required to have certain chemical and physical properties as measured by particular tests. Briefly stated, the four types consist of three groups of 100% rag fiber papers and one group of chemical wood pulp fibers, each group with varying degrees of whiteness, translucency, strength, and permanence. The general requirements are that color does not change, according to subjective comparison, after accelerated aging (100 degrees C, 72 hours); that opacity averages, depending on classification type, from 28-70% and not increase more than 3-7% for rag papers or 9% for chemical woodpulp; and that fold endurance not drop by more than 50% after aging. Manufacturers have tried to meet these standards, despite inherent vagueness (i.e. specifying ill-defined 100% "rag" rather than cotton linters or alpha cellulose; subjective color "measurement") and the recent modification requiring minimum inclusions of 25-50% "recovered material" in rag and chemical wood pulps respectively.

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