

Abstract

Using information derived from a study characterizing types of pigment-coated papers, the authors investigated the effects of various treatments on selected examples. To evaluate the effects of some solvent application techniques on pigment-coated papers, two projects were designed. The first project was a preliminary investigation, testing papers hand-coated with traditional recipes, to subjectively compare the effects of different stain removal solvents (water, ethanol, acetone, and toluene) and application techniques (poultice, immersion, and suction disk). Findings, based on SEM imaging, indicate that some application techniques and some solvents can cause microfissures or loss in coating material. The second project was a statistical study using contemporary machine-coated papers to provide reproducible data on the effect of two treatments which have been used for cleaning dirt, stains, or accretions from pigment-coated papers, namely aqueous immersion and blotter-poulticing, followed by air- or blotter-drying. Findings evaluated changes detected by SEM imaging and measured by color (CIE L*a*b*) and gloss (85° angle). SEM imaging revealed more surface disruption in blotter-poulticed and blotter-dried samples. Immersed and air-dried samples underwent a greater unit reduction in gloss than poulticed, blotter-dried samples. The glossiest paper underwent the greatest unit reduction in gloss following all treatments.

Keywords

Coated papers, prepared grounds, chromolithographs, solvent applications, poultice, aqueous immersion, drying, stain removal, accretion removal, color, gloss, technology, history, treatment trials, research design

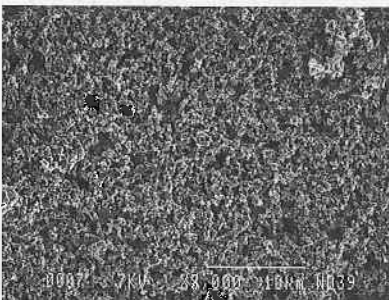


Fig. 1a: Hand-coated paper with calcium carbonate:Liquitex rabbit skin glue (1:3) applied in 5 coats

Pigment-Coated Papers II: The Effects of Some Solvent Application Techniques on Selected Examples

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Introduction

Pigment-coated papers can pose particular problems for collections and conservators because of their special formulations. Such formulations, which were the focus of an earlier study, render pigment coatings especially susceptible to damage from abrasion, flaking, fingerprint and oil stains, water stains and blocking, and adhesive and tape accretions and staining [1-5]. Ironically, their special formulations also render the pigment coatings susceptible to damage from some of the conservation treatments which might be used to correct the problems noted above. This second study was developed to evaluate some of the effects of various application techniques of solvents on selected examples of pigment-coated papers. It consists of two projects described below, following a brief overview of treatment research and design.

Treatment Research and Design

Treatments for pigment-coated papers, published in the conservation literature, have included the use of organic and aqueous solutions applied by steam, brushes, and poulticing for the removal of stains and accretions [3,4,5,7,8]. Similar treatments used by the authors have been found to be very successful, although microscopic examination has indicated that organic and aqueous solutions may cause the break up, microfissuring, or loss of pigment coatings (see Figs. 1 and 2)[1,8]. Consequently, two treatment projects were developed, as indicated in the research design.

Research design for pigment-coated paper projects.

A. Characterize and select pigment-coated paper samples for treatment testing (hand-coated and machine-made pigment-coated papers).

B. Select treatment procedures to be tested.

Project 1: Effects of stain removal solvents & application techniques on hand-coated papers.

Water	Ethanol	Acetone	Toluene	Untreated control
Application techniques:				
Immersion		Poultice	Suction disk	

(Note: For each paper type each sample was tested once.)

Project 2: Effects of accretion removal techniques on machine-coated papers.

						Untreated control		
Immersion			Poultice			C1	C2	C3
A1	A3	A3	B1	B2	B3			
Air Blot	Air Blot	Air Blot	Air Blot	Air Blot	Air Blot			

(Note: For each paper type each sample under categories A1-C3 was tested 3 times.)

C. Evaluate general findings based on changes in appearance (SEM imaging) and/or properties (color/brightness and gloss).

The first project was a preliminary study using SEM imaging to compare the effects of solvents (water, ethanol, acetone, and toluene) and application tech-

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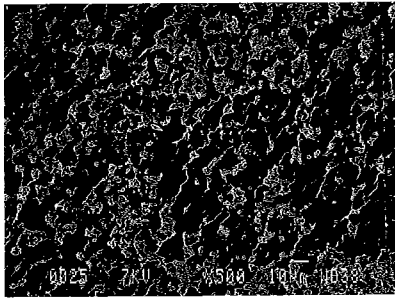


Fig. 1b: After hand-burnishing

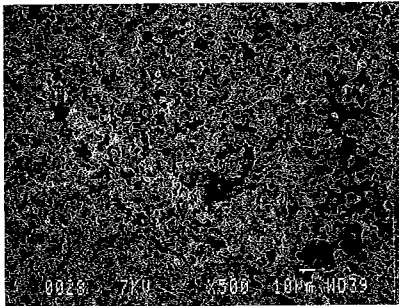


Fig. 1c: After application acetone by suction table to lower left corner; shows break-up of burnished surface

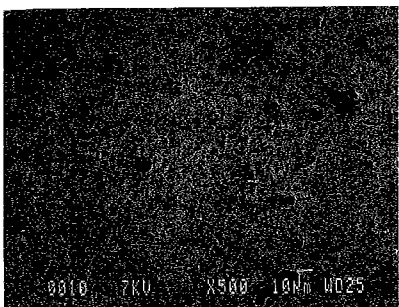


Fig. 2a: Hand-coated paper with zinc oxide:Liquitex rabbit skin glue (2:3) applied in 5 coats

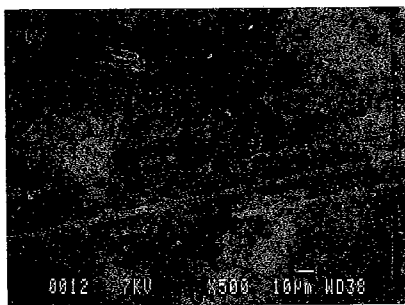


Fig. 2b: After hand-burnishing

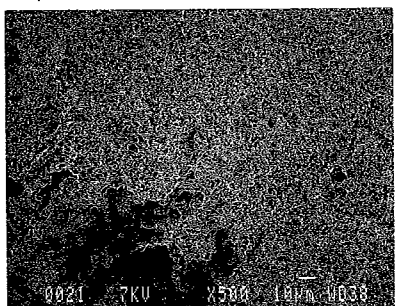


Fig. 2c: After application acetone by immersion to lower left corner; shows break-up of burnished surface

niques (poultice, immersion, and suction disk), used in conservation for stain removal, on traditionally hand-coated papers. The second project was a statistically controlled study comparing the effects of accretion removal techniques (aqueous immersion and blotter-poulticing) on contemporary pigment-coated (machine-coated) papers. Changes in appearance of the pigment coating with respect to surface structure were observed by SEM imaging; brightness was measured by colorimetry; and gloss was measured by a glossmeter. The following summaries for each of the two projects describes the A) paper samples characterized, B) treatment procedures used, and C) general findings.

Project 1: Effects of stain removal solvents and application techniques on hand-coated papers

A) Paper Samples Characterization: A survey of traditional recipes provided the basic information for sample preparation [9]. The sample papers were hand-coated with combinations of calcium carbonate, zinc oxide and barium sulfate in binders of gum, glue, and acrylic. A machine-coated paper composed of barium sulfate and calcium carbonate in an acrylic binder was examined and tested as a comparison exemplifying a contemporary pigment-coated paper. Six test papers were prepared by hand-brushing a paper support (machine-made wove paper of high alpha cellulose rawstock) with one of each of the following formulas, made with reagent grade compounds:

1. calcium carbonate:Liquitex rabbit skin glue (1:3) applied in 5 coats
2. calcium carbonate:gum arabic (1:2)
3. zinc oxide:Liquitex rabbit skin glue (2:3) applied in 5 coats
4. zinc oxide:gum arabic (2:3)
5. zinc oxide:Liquitex gel acrylic medium
6. barium sulfate and calcium carbonate : Liquitex gel acrylic medium

B) Treatment Procedures: Four solvents (water, ethanol, acetone, and toluene) were applied by three techniques (immersion, poultice, and suction disk) to each sample of paper using the techniques described below:

Immersion technique: For the immersion technique, the end of each sample was dipped into a 3 mL deep solution of solvent and held there for 3 seconds. The sample was then removed and allowed to air-dry.

Poultice technique: The poultice technique consisted of placing diatomaceous earth saturated by solvent (approximately 1-2 mL solvent to 0.3 grams diatomaceous earth, depending on solvent) on the front of each paper sample lying on a non-absorbent support.¹ The poultice covered an area approximately 5mm in diameter, and contrary to normal practice, the wet poultice was not surrounded by dry poultice to diffuse the transition from wet to dry areas. The poultice was allowed to air-dry before being removed by an air bulb and brushing.

Suction Disk technique: The suction disk technique consisted of applying three drops of each solvent locally by dropper on a 15 cm fritted glass bead disk (masked off with polyester film), which can reach a pressure of c. 25" Hg.

C) General Findings: SEM imaging (described below) indicated that certain application techniques caused changes to some papers. For instance, acetone applied by suction table cause the break-up and/or loss of the burnished surface of a paper hand-coated with calcium carbonate and Liquitex rabbit skin glue (Fig. 1). A similar change occurred when acetone was applied by immersion to a paper hand-coated with zinc oxide and Liquitex gel acrylic medium (Fig. 2). The aqueous poultice applications used in this study also caused microfissuring of some pigment-coated surfaces, especially the machine-coated sample [1,4,9].

Project 2: Effects of selected accretion removal techniques on contemporary machine-made pigment-coated papers

A) Paper Sample Characterizations: Several contemporary machine-made pigment-coated papers were selected and assigned codes such as those in quotes below. The papers were supplied by various (anonymous) manufacturers, whose accompanying literature described the papers as follows:

- "B": Machine-made in France as "Special Point d'Argent: Calligraphie" (recommended by manufacturer for silver point drawings).
- "C": Machine-made 100% rag paper with neutral pH. Smooth, clay-coated; recommended for silverpoint drawing.
- "F": Machine-made glossy, light weight stock, available in 28 colors.
- "K": Glossy white text-weight paper recommended for illustrations and ink drawings.
- "L": Machine-made in Germany; smooth, glossy surface on 2 ply, 10 point card stock.
- "Q": Machine-made with fine clay coating. Ivory color, recommended for silverpoint drawing.
- "R": Machine-made 100% rag paper with neutral pH. Smooth, clay-coated (2 sided); glossy on one side and matte on the other. Recommended for off-set printing but not silverpoint drawing.
- "S": Machine-made paper with smooth clay coating recommended for silverpoint drawing.

The machine-coated papers were also characterized in-house by analysis of pigments and binders using the following instrumentation techniques and measurements:

SEM: SEM imaging of coating surface and cross-section morphology, and SEM/EDS analysis of pigments, were carried out on a Jeol JXA - 840 A scanning electron microscope with Tracore Northern TN 5502 energy dispersive x-ray analysis system. The samples were mounted on aluminum stubs and gold coated for imaging, and on carbon stubs and carbon coated for elemental analysis.

FTIR: FTIR analysis of binders was carried out on a Mattson Cygnus 100 Fourier Transform Infrared Spectrophotometer with a Spectratech IR-Plan Microscope. Surfaces of coated papers were analyzed by reflectance, or alternately micro samples of coatings were removed and pressed into thin films in a diamond anvil cell for analysis by transmission.

Colorimetry/Brightness: Color (specular reflectance included) of coatings was measured with the HunterLab Ultrascan Spectrocolorimeter (D_{65} , 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). Three measurements were taken per sample and averaged. Brightness was calculated for the untreated papers by the following equation: $B = 0.01L^2 - bL/70$. Ultrascan measurements are comparable to GE Brightness used by industry (see Tables in previous article), which measure reflectance at 457nm using a tungsten source, except that Ultrascan measurements will be higher when optical brighteners are involved.

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

The elemental compositions of the contemporary machine-coated papers are listed in Table I. This was compared with the previous article's Table I of the elemental composition of common coating pigments. The earlier table indicates the refractive index and general size of pigment particles as used in the paper coating industry, which along with particle shape contribute to the brightness, gloss, and opacity of the final coat. For instance, refractive index (n) influences a pigment's ability to refract light, contributing to opacity, which is important for reducing print show-through in contemporary papers. Size (microns or μm) also affects a pigment's ability to refract and reflect light; as the particle size decreases, opacity increases [10]. Particle size also affects surface smoothness and contributes to gloss, depending on porosity, compactness, etc. A size of 0.12 may be best for ink holdout, while 0.2 might be best for gloss, but 0.5 may have the best combination of gloss and light scatter [11].

Table I. Elemental composition of selected machine-coated papers based on SEM/EDS.

Papers	Al	Si	K	Ti	Ca	S	Ba
"B"	-	+	-	-	+	+	+
"C"	+	+	-	+	-	-	-
"F"	+	+	-	+	+	-	-
"K"	+	+	+	-	+	+	-
"L"	+	+	+	-	+	+	-
"Q"	+	+	-	+	+	-	-
"R"	+	+	-	+	+	-	-
"S"	+	+	-	+	+	-	-

Based on the comparison of elemental compositions shown in both Tables I (of this and the previous article), the contemporary machine-coated papers can be divided into three groups. The first group ("B") is made primarily of barium sulfate (Ba, S), perhaps with talc (Si, Ca) or gypsum (Ca, S). The second group ("K" and "L") contain primarily clay (Al, Si, K) and gypsum (Ca, S). The third group (the remainder) contain primarily clay (Al, Si) and talc (Ti, Ca) or titanium dioxide and calcium carbonate. The pigment-coating compositions of these papers were compared with the composition of several pigment-coated chromolithographs undergoing contemporaneous conservation treatment [6]. Based on these comparisons, one paper from each of the three groups was chosen for treatment trials and testing. The three selected contemporary machine-coated papers, designated by the codes "B," "S," and "L," are described in Table II. By comparing the information in Table II of this article with the information

Table II. Characterization of selected machine-coated experimental papers, with Ultrascan brightness (converted from L* spectral reflectance value) and Reflektometer gloss (at an 85° angle).

Papers	Rawstock (estimated percentages)	Pigment composition		Binder (FTIR)	Bright- ness (Hun- ter)	Appearance	85° gloss
		SEM/EDS	FTIR				
"B"	100% ground hardwood and softwood (estimate)	Ba, S, Ca	sulfate, some clay	protein, resin	91.66	matte, smooth	18.5
"S"	40% chemical, 50% mechanical wood pulp, 10% rag (est.)	Ti, Si, Al, Ca	clay, calcium carbonate	styrene acrylic resin	85.08	matte, slightly rough	11.8
"L"	85% chemical softwood, with some chemical hardwood, 15% rag (est.)	Si, Al, Ca, S, K	clay	protein, acrylic	89.65	very glossy, smooth	96.9

in Table II of the previous article, the respective grades of the papers may be estimated. For instance, the make-up of paper "B" suggests it may be a lower grade paper (Grade 4 or 5); "S" may be a middle grade (Grade 2 or 3); and "L" may be a higher grade (Grade 1).

B) Treatment Procedures: The treatment procedures compared effects of water applied by two techniques that might be used to remove accretions or to humidify a piece prior to flattening. The two treatment techniques were immersion and poulticing with a damp blotter. These two treatments were selected to represent

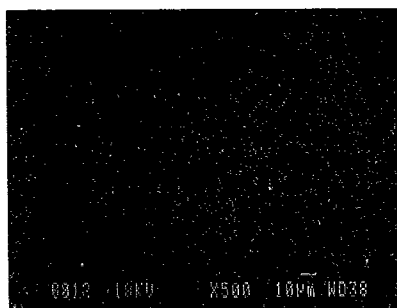


Fig. 3a: Machine-coated paper "B" before treated

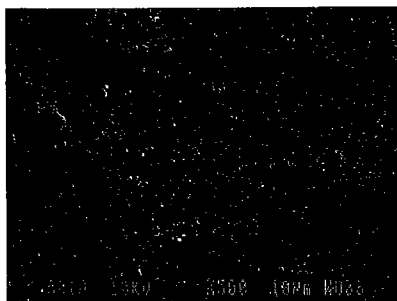


Fig. 3b: After immersion and air-drying

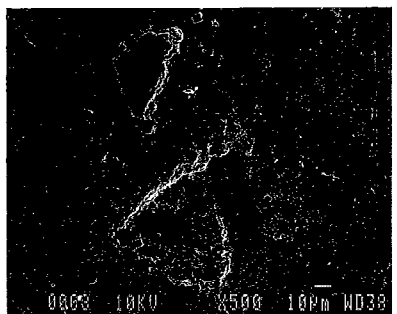


Fig. 3c: After blotter-poulticing and blotter-drying

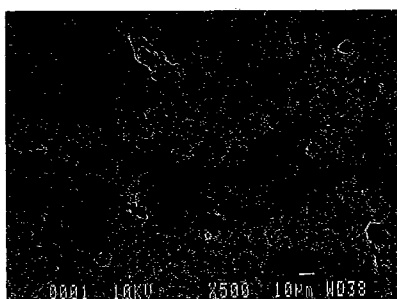


Fig. 4a: Machine-coated paper "S" before treated



Fig. 4b: After immersion and air-drying

differences in amount of solvent absorption, exposure time to solvent, and direction of application and evaporation. For example, aqueous immersion may result in greater penetration, longer exposure, and more lateral movements of compounds as compared to aqueous poulticing. Half of the samples were exposed to a five minute immersion treatment in deionized water, while the other half of the samples were poulticed with a damp blotter for five minutes. Half of each set of the treated samples were air-dried and the other half were dried in a blotter press. The experimental conditions are outlined in Appendix A, with details of the procedures as follows:

Immersion Procedure: The samples were immersed in deionized water on a polyester web (nonwoven, with a smooth surface, 5 mil) on a polypropylene screen. The samples remained immersed for five minutes. The screen with web and samples was lifted out of the water and allowed to drain for two minutes. Half of the samples were lifted on the polyester web to a unbuffered blotter on a felt and were allowed to air-dry totally. Appearance measurements (color and gloss) were taken after two days. The other half of the samples were placed onto an unbuffered blotter on a felt and allowed to air-dry for 5–10 minutes until all standing water on the surfaces of the papers evaporated. The unbuffered blotter under the samples was replaced by a dry unbuffered blotter and these samples were then covered with polyester web, unbuffered blotter, and felt, and were blotted with hand pressure. Both top and bottom unbuffered blotters were changed and the ensemble was placed for five minutes in a "press" under a felt and $\frac{1}{2}$ inch thick piece of plexiglass (less than 1 PSI). The unbuffered blotters were changed again after one hour and returned to the "press". The polyweb and unbuffered blotter were changed the next day and returned to the "press." Appearance measurements (color and gloss) were taken after two days.

Poultice Procedure: The unbuffered blotter used for poulticing was dampened by immersing it on a screen in a tub of deionized water. The screen was removed and the unbuffered blotter was drained on the screen for two minutes. A piece of 4 mil polyester film was placed on the unbuffered blotter resting on the screen and then the unbuffered blotter was flipped over so that the polyester film was on the bottom. The unbuffered blotter was allowed to sit for five minutes to enable standing water to evaporate for more even moisture distribution. The unbuffered blotter was then flipped over onto the samples which were against a formica countertop and the polyester film was removed. A felt and a $\frac{1}{2}$ inch thick piece of plexiglass were placed on the unbuffered blotter-poultice for 5 minutes. After the plexiglass, felt, and damp unbuffered blotter were removed, the wet samples were exposed to the air for two to five minutes, until all standing water on the surfaces of the papers evaporated. Half of the samples were left to air-dry totally. Appearance measurements were taken after two days. The other half were moved to a blotter "press" made up, from the bottom up, of the formica counter, felt, unbuffered blotter, polyester web, sample, polyester web, unbuffered blotter, felt, plexiglass (less than 1 PSI). The samples were left overnight in the press. The next day the polyester web and unbuffered blotter were changed and the ensemble was returned to the "press." Appearance measurements were taken after two days.

C) General Findings: Changes in the papers after treatment were assessed by SEM imaging and by measuring optical properties of color and gloss, as follows:

SEM: All papers were found to have undergone surface changes following all treatments when viewed with a scanning electron microscope. As compared to the untreated controls, immersed air-dried samples generally showed less change than the poulticed blotter-dried samples (Figs. 3–5). For the barium containing paper "B," the immersed samples generally exhibited raised areas and microfissures. In comparison to the untreated control (Fig. 3a), the immersed air-dried samples showed fibers more exposed (Fig. 3b), while the immersed blotter-dried samples showed disturbed areas and apparent lifting of the surface. The poultice air-dried samples showed a disturbed surface characteristic of lifting and delamination, while the poultice blotter-dried sample showed microfissuring, lifting of coating, and wrinkles (Fig. 3c). For paper "S," the immersed samples were found to have a more pitted surface with more exposed fibers as compared to

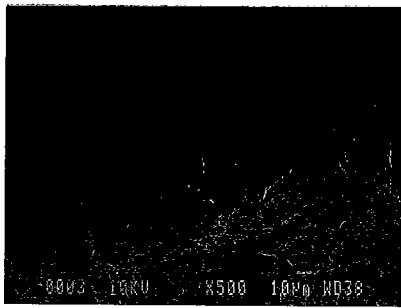


Fig. 4c: After blotter-poulticing and blotter-drying

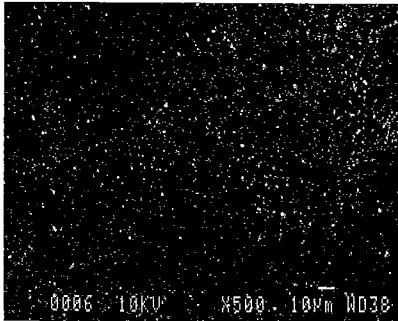


Fig. 5a: Machine-coated paper "L" before treated

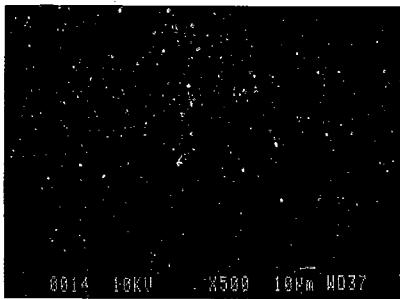


Fig. 5b: After immersion and air-drying

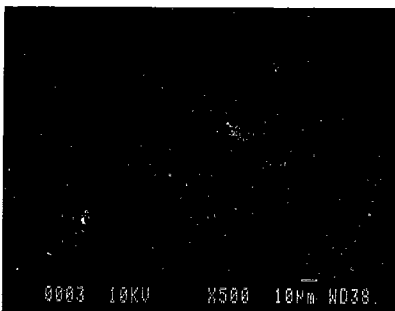


Fig. 5c: After blotter-poulticing and blotter-drying

the untreated control (Fig. 4a). The immersed air-dried samples also exhibited microfissuring (Fig. 4b), while the poulticed blotter-dried samples had microfissures and depressions (Fig. 4c). For glossy paper "L" small holes or microfissures were found in all treated samples (Figs. 5a-c).

Colorimetry: All of the papers exhibited darkening (decrease of L^* or brightness) and yellowing (increase in b^* , Fig. 6). However, the changes are all less than one unit and could not be considered visually significant, regardless of statistical significance. Greater darkening (and yellowing) occurred with the immersed air-dried samples, followed generally by the immersed blotter-dried samples.

Gloss: All samples showed a significant reduction in all gloss measurements regardless of whether the measurement was made at an angle of 20, 60, or 85 degrees. Gloss reduction tended to be greatest following immersion and following air-drying (Fig. 7). The greatest change was measured at 85 degrees and for

Fig. 6: CHANGES IN COLOR (b^*)

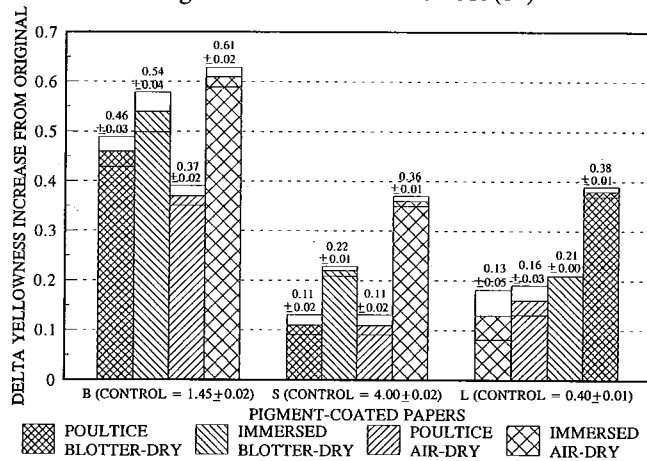


Fig. 6: Colorimetry

Fig. 7: CHANGES IN GLOSS (85°)

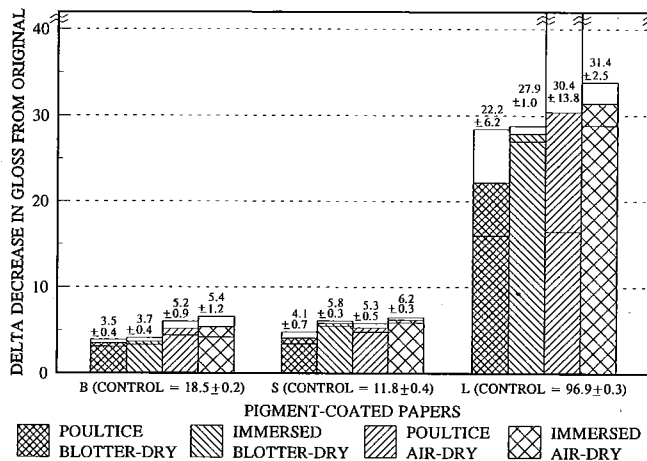


Fig. 7: Gloss

the glossiest paper ("L"); standard deviations were large for this paper, especially for the poulticed measurements. This suggests that the gloss changes to this paper were not uniform. (See Appendix B for gloss measurements).

Conclusion

With respect to solvent application techniques, SEM imaging indicated that specific application techniques could cause changes in the surfaces of some papers. For instance, acetone applied by suction table caused the break-up, microfissuring, and/or loss of the burnished surface of a paper hand-coated with calcium carbonate and Liquitex rabbit skin glue (Fig. 1). A similar change occurred when

acetone was applied by immersion to a paper hand-coated with zinc oxide and Liquitex gel acrylic medium (Fig. 2). The aqueous poultice applications used in this study also caused microfissuring of some pigment-coated surfaces, especially the machine-coated sample [1,4,8].

Aqueous immersion and blotter-poulticing, followed either by air-drying or blotter-drying, of three different types of contemporary machine-made, pigment-coated papers, caused changes in surface appearance and significant reductions in gloss and color/brightness (increased darkening and yellowing). The greatest structural changes, as observed by SEM imaging, occurred in the blotter-poultice samples, especially for less glossy papers. The greatest unit reduction in gloss, as measured instrumentally, occurred in the glossiest paper and was most irregular for the poulticed samples. Findings suggest that while aqueous blotter-poulticing and blotter-drying may be appropriate for some pigment-coated papers, other procedures might cause less surface change to highly glossy coated papers.

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Note

1. Diatomaceous earth (hydrated silica from diatom plant skeletons) was selected as a poultice 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.

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Appendices

Appendix A. Experimental conditions and labelling. Each treatment trial was conducted three times and each resultant sample was measured three times for statistical accuracy.

Contemporary pigment coating	S	L	B
Control (C)	1 = C	14 = C	27 = C
Immerse/airdry (IAD)	2 = IAD.1 (3 meas)	15 = IAD.1 (3 meas)	28 = IAD.1 (3 meas)
	3 = IAD.2 (3 meas)	16 = IAD.2 (3 meas)	29 = IAD.2 (3 meas)
	4 = IAD.3 (3 meas)	17 = IAD.3 (3 meas)	30 = IAD.3 (3 meas)
Immerse/blotter (IBD)	5 = IBD.1 (3 meas)	18 = IBD.1 (3 meas)	31 = IBD.1 (3 meas)
	6 = IBD.2 (3 meas)	19 = IBD.2 (3 meas)	32 = IBD.2 (3 meas)
	7 = IBD.3 (3 meas)	20 = IBD.3 (3 meas)	33 = IBD.3 (3 meas)
Poultice/airdry (PAD)	8 = PAD.1 (3 meas)	21 = PAD.1 (3 meas)	34 = PAD.1 (3 meas)
	9 = PAD.2 (3 meas)	22 = PAD.2 (3 meas)	35 = PAD.2 (3 meas)
	10 = PAD.3 (3 meas)	23 = PAD.3 (3 meas)	36 = PAD.3 (3 meas)
Poultice/blotter (PBD)	11 = PBD.1 (3 meas)	24 = PBD.1 (3 meas)	37 = PBD.1 (3 meas)
	12 = PBD.2 (3 meas)	25 = PBD.2 (3 meas)	38 = PBD.2 (3 meas)
	13 = PBD.3 (3 meas)	26 = PBD.3 (3 meas)	39 = PBD.3 (3 meas)

Appendix B. Data: Gloss, 85 degrees (standard deviations).

	S	L	B
Absolute			
Control (C)	1 = 11.8 ± 0.4	14 = 96.9 ± 0.3	27 = 18.5 ± 0.2
Delta			
Immerse/airdry (IAD)	2-4 = 6.2 ± 0.3	15-17 = 31.4 ± 2.5	28-30 = 5.4 ± 1.2
Immerse/blotter (IBD)	5-7 = 5.8 ± 0.3	18-20 = 27.9 ± 1.0	31-33 = 3.7 ± 0.4
Poultice/airdry (PAD)	8-10 = 5.3 ± 0.5	21-23 = 30.4 ± 13.8	34-36 = 5.2 ± 0.9
Poultice/blotter (PBD)	11-13 = 4.1 ± 0.7	24-26 = 22.2 ± 6.2	37-39 = 3.5 ± 0.4

ICOM COMMITTEE FOR CONSERVATION

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