Abstract

Pigment-coated papers have been used, virtually since the beginning of papermaking history, for their special properties. These properties, however, may render coated papers more susceptible to certain types of damage and more reactive to certain conservation treatments. This article, the first of two, provides a chronology of the history and use of coating materials and techniques, with an emphasis on the roles that pigment properties play in influencing the final coating properties. Some common pigments used for paper coatings are listed, along with their elemental compositions and properties such as particle size, refractive index, brightness, and pH. The influences of various binders (protein, starch, resin and synthetics), additives (eveners, dispersing agents, anti- and defoamers), and finishing techniques (supercalendering) on coating properties are also outlined. U.S. grading standards for modern pigment-coated papers, based on rawstock, brightness (single wavelength reflectance), and gloss (75° angle), are listed to aid in the characterization of unknown papers.

Keywords

Coated paper, pigment-coated paper, clay-coated paper, color-coated paper, prepared paper, prepared-ground paper, enamel paper, surface-sized paper, hand-coated paper, brush-coated paper, machine-coated paper, off-machine coated paper, brightness, gloss, technology, history, treatment

Pigment-Coated Papers I: History and Technology

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Introduction

Museums, libraries, and archives contain large collections of pigment-coated papers, which are often the primary support for various drawings and printing processes. These processes are executed on papers having opaque white pigment coatings because of the special properties imparted by such coatings. For instance, pigment-coated papers provide a smooth, white ground for metal point drawings and ink illustrations; an inert ground capable of maintaining registration throughout multiple inkings for chromolithographs or off-set lithography; and a luminous, sharp background for letterpress, relief, intaglio, and photomechanical prints, bookplates, and photographs.

Unfortunately, pigment-coated papers may be more easily marred by scratches, and more retentive of stains and dirt, than uncoated papers [1–9]. This is because pigment-coated papers are produced by modifications to standard papermaking practices with respect to furnish (materials such as rawstock, pigments, binders, and additives) and formation (manufacturing procedures such as calendering) [10–32]. Such modifications are necessary to achieve the specific properties of brightness, gloss, smoothness, opacity, and ink receptivity required, in particular, by the printing trade. The various formulation materials and techniques, and their effects on the properties and use of pigment-coated papers, are the subject of this article, which is the first of two studies on the topic. The special problems posed by pigment-coated papers requiring conservation treatment involving the application of solvents is the subject of a second study reported in these Preprints.

Numerous terms are used to describe pigment-coated papers. Generally, the terms reflect the diversity of materials and techniques used to coat papers. For instance, such papers may be described as coated paper, pigment-coated paper, clay-coated paper, color-coated paper, prepared paper, prepared-ground paper, enamel paper, or surface-sized paper, etc., which may be one-side coated, two-side coated, light weight coated, hand-coated, brush-coated, machine-coated, or off-machine coated, etc. The following review of the literature attempts to define some of these terms and provide a brief historical overview of materials and techniques used in formulations. All dates noted in this section should be interpreted as approximate, since dates may vary depending on reference and geographical sources, or change as new information is discovered by future research.

Formulations: some materials and techniques used up to the mid-20th century

Pigment-coated papers consist of a paper base covered by a mixture containing at least a binder and inert pigment (Table I). The earliest examples may have originated from China, where paper surviving from 450 CE is reported to have a starch sizing and gypsum surface treatment [16]. During the Tang Dynasty (CE 618-907) papers may have been coated with white mineral powders and wax, which filled interstices between fibers in order to increase water repellency and smoothness for fine calligraphy [20]. By the 8th century, Arab cultures had reportedly modified paper with talc, gypsum or chalk, which could also be mixed with rice starch to coat the paper for increased whiteness [10, 12].

During the middle ages, papers were coated with white pigments to provide an appropriate surface for metal-point drawings, executed with a metal stylus made of silver or copper, which before the discovery of graphite represented the only

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Table 1. Some typical coating pigments with common names and commercial use, as well as elemental composition and relative properties of refractive index (n), average optimum particle size in microns (μm), brightness (GE percent reflectance at 457 nm), and pH. [Compiled from Beazley, Casey 1961, Kourik.]

Pigments	Al	Si	K	Ti	Ca	S	Ba	Zn	Pb	n	μm	Brightness	pH
Barium sulfate (baryta or blanc fixe) {weight, purity, brightness, finish}	–	–	–	–	–	+	+	–	–	1.64–1.66	0.11–0.54	95–98	3.5
Calcium carbonate (ground or precipitated) {opacity, brightness, dull finish, ink affinity}	–	–	–	–	+	–	–	–	–	1.57–1.66	0.1–10	93–98	7–10
Calcium sulfate (gypsum or pearl white, crown filler, alabastine, etc.)	–	–	–	–	+	+	–	–	–	1.52	0.2		
Calcium sulfite {brightness, absorbency}	–	–	–	–	+	+	–	–	–		6.5	92–96	8–9
Clay (residual or transported, kaolin) {body, finish}	+	+	–/+	(+)	–	–	–	–	–	1.55–1.56	2.0 0.4–5.0	65–91	4.3–7
Diatomaceous silica {dull, abrasive, resistant to blackening}	–	+	–	–	–	–	–	–	–		1.5 × 10–50	65–95	5
Lead white (basic lead carbonate, hydrocerussite)	–	–	–	–	–	–	–	–	+	1.80–2.08			
Satin white (calcium sulfoaluminate) {finish, brightness, gloss}	+	–	–	–	+	+	–	–	–	1.46	1–2 by 0.1–0.2		
Talc	(+)	+	–	–	(+)	–	–	–	–	1.57	2.0		4–6
Titanium dioxide (rutile or anatase) {opacity, brightness, whiteness, smoothness}	–	–	–	+	–	–	–	–	–	2.5–2.7	0.2–0.5	97–98	4–6
Zinc sulfide	–	–	–	–	–	+	–	+	–	2.37	0.3	94–96	
Lithopone (Orr's White, Charlton White)	–	–	–	–	–	+	+	+	–	1.84–2.00	0.3–0.5		
Zinc oxide (opacity)	–	–	–	–	–	–	–	+	–	2.01–2.03			
Polystyrene	–	–	–	–	–	–	–	–	–	1.59	0.5		

"dry" medium available for very finely detailed drawings [2]. Coated or prepared-ground papers had the additional advantage of being "erasable," in that media could be scraped and/or burnished away (or coated over) if changes were desired. To prepare such paper, coating material, mixed with approximately two parts water, was poured on and hand-brushed evenly over the paper, which was then loft-dried [23]. Burnishing, usually by hand with agate stone, created different surface effects, such as increasing smoothness by evening out surfaces, saturating color by compressing pigment, and creating luminosity from increased gloss. Early treatises on recipes for prepared papers mention ground bone ash, powdered cuttlebone, or gypsum, mixed with aqueous binders of glue or gum, or with linseed oil [15, 19, 24, 29]. A treatise on Persian paintings reportedly suggests a two-layered ground consisting of plaster, glue and grape treacle topped by lead white and oil varnish [7].

By the 16th century, coatings were applied to the backs of playing cards, to improve strength and durability. By the mid-16th century (1540) hammer-glazing had replaced hand-burnishing [23].

In the mid-18th century (1764), a coating mixture containing lead white, plaster of Paris, lime size, and nut or linseed oil was patented [19]. There is some reported use at this time of zinc white (1775), a very white, bright and opaque pigment [21, 22]. After the turn of the century, in 1827, enamel paper was made with lead white, isinglass, gum, and animal size, several coats of which were applied and then burnished by plating or running the coated paper, in contact with a steel plate, through a press [19, 22]. By 1830, high gloss could also be imparted by burnishing wax and rosin-treated paper with calendering [19].

The early 19th century witnessed the development of several new coating mixtures. The most popular mixture, introduced around 1807, was a combination of casein and animal glue with China Clay or kaolin (hydrated aluminum silicate extracted from feldspar in granite, with traces of muscovite mica and potassium in primary or residual sources, or yellowing titano-ferrous impurities in secondary or transported sources) [12]. Clay coating made paper whiter, heavier and more receptive to ink. The end of the century (1879) marked the introduction of a pigment coating used for the production of high gloss art and offset printing paper, satin white (from slaked lime treated with paper makers' aluminum sulphate). Satin white has a high pH, enabling it to augment clay, and to be used in binders of gum arabic, casein, soy protein, hydroxyethylated starch, or carboxymethyl cellulose to provide a satin-like gloss following relatively light calendering [21]. It also has a bright color, but since it is expensive, difficult to handle, and has a high adhesive demand, it is used today primarily for off-machine coating of specialty papers [14]. By the end of the 19th century there was an increased use of baryta (specifically precipitated barium sulfate from barite). Barium sulfate itself comes in two forms, either as the natural mineral barite or as the artificial blanc fixe which may be derived from barite or witherite (a barium carbonate ore in England and Europe). Baryta, or blanc fixe, makes a brighter, denser, and less porous coating, used for special grades of chart papers and for photographs, although it is now being replaced in part by titanium dioxide [14, 21]. When barium sulfate is co-precipitated 7:3 with zinc sulfide, the combination forms lithopone, which became a substitute for lead carbonate. Another, lesser used, pigment was calcium carbonate (natural ground chalk and limestone from calcite, or precipitated as aragonite).

The 20th century ushered in several new materials, starting with the introduction in 1906 of titanium dioxide (made from ilmenite, a double oxide of titanium and iron, processed by sulfuric acid or chlorine, and occurring as either anatase or the more opaque rutile). Titanium dioxide is so opaque and white that only a small amount is needed [21]. It may be bulked up by calcined clay or substituted 25-50% by synthetic silicas and silicates [12, 21]. Also used during the early part of the century were binders of rubber latex (1927), peanut and soy proteins (1937), and pigments such as precipitated calcium carbonate (1925), diatomaceous earth (1938), and zinc oxide (1933), used for early photocopying paper. By the middle of the century, fluorescent whitening agents (stilbenes, salicylates, benzophenones, benzotriazoles and others) [3, 21, 26], and acrylic gessos [8] had been developed, and there was more use of talc (hydrated magnesium silicate with hydrophilic, hydrophobic, and oleophilic properties), used to control wax-like pitch from sulphite wood pulp [21]. Resin binders included urea and melamine formaldehyde resins (1946), polyethylene resins (1946), acrylic emulsions (1952), acrylonitrile and polyvinyl alcohols [9], as well as latexes of rubber, butadiene-methacrylate and styrene-butadiene (1947) [21].

The 19th and 20th centuries also fostered the development of new coating application techniques. In the last quarter of the 19th century, as hand-made paper was replaced by machine-made paper, hand-coating and hand-brushing were replaced by automated "off-machine" coating processes. Off-machine coating is carried out separately from the papermaking machine. One of the earliest examples employed a single-surface brush-coating machine (1852) which used a splatter brush to apply to the paper the correct weight of coating material which was then smoothed by a coarse brush, refined by a firm brush, and finished by badger-hair brushes, and festoon-dried [6]. This application technique resulted in poor uniformity of coating when tested by quality-control measurements of

"wet thumb" and "thumb rub" tests [23]. By the late 19th century (1880), double-surface brush-coating processes were developed. Since brush maintenance was time-consuming and expensive, brush-coaters were replaced at the turn of the century by roll-spreading coaters (1920), which quickly and evenly metered and spread coating material on paper using rotating rolls. Roll-coaters worked best with starch adhesives. These early high-speed coaters initiated the eventual mass production of coated papers [13]. Toward the middle of the 20th century air-knife coaters (1940) were developed, which used a stream of air from a transverse nozzle to control both coating weight and spread in one simple operation, by leveling off excess coating to form a smooth surface which was dried in hot air tunnels. Later developments resulted in the flexible or trailing blade-coater (1950), which utilizes a flexible blade running against paper backed by a rubber roll, followed by drying in air cap driers. More recent developments include extrusion coating, whereby thermoplastic polyethylene resin is melted and extruded in a continuous flow under light pressure through a film-forming slot-die; it is then stretched and rapidly combined with paper between a rubber-covered pressure roll and a water cooled chromium plated steel roll to form a lasting bond with the paper [21].

Formulations: some current materials and techniques

Today, pigment-coated papers account for 20% of all paper sold in the United States. At least ten types of coaters have been developed, including dip, brush, knife, roll, air-brush, spray, extruded, print, cast, and strip coaters. Currently, most coated paper is machine-made, meaning that the coating is applied to the paper as part of the papermaking process. Coated paper may be graded in different ways, including by use or by function, such as coated litho grade (water-resistant sized, one-sided coated paper) or coated book grade (suitable for reproducing halftone photomechanical illustrations) [13, 14]. Examples of U.S. paper grade standards for pigment-coated papers are outlined in Table II.

Table II. Examples of grading standards in the U.S., with GE brightness (percent reflectance at 457 nm) and TAPPI gloss (T480 at a 75° angle). [Sources: Casey 1961, 1985.]

Grades	Types	Uses	Rawstock	Brightness (GE)	Gloss 75° (separate grading system)
1	Merchants Grade, coated enamel, coated woodfree	multicolor printing, advertising, brochures	100% chemical pulp (basis weight g/m² = 100–150)	85+	45+ (glossy)
2	Merchants Grade, coated enamel, coated woodfree	multicolor printing, advertising, brochures	100% chemical pulp (basis weight g/m² = 75–150)	83–84.9	25–35 (dull)
3	magazines, catalogs	magazines, catalogs	chemical & mechanical	79–82.9	25– (matte)
4	coated groundwood	magazines, catalogs	mechanical & chemical	73–78.9	25– (matte)
5	publication grade, coated groundwood	magazines, newspapers	mechanical	72.9–	25– (matte)

Pigments

Modern coatings are composed not only of pigments and binders, but also additives and fluid (water). Ninety percent of today's pigment-coated papers are clay coated. The size of clay particles varies greatly and affects covering power, brightness, gloss potential, and ink and varnish hold-out. Calcium carbonate is

currently the second most important pigment, and it is used with clay to increase brightness, opacity and ink receptivity. Some pigments have been put to new uses. Amorphous silicas and silicates, such as diatomaceous silica (Kieselguhr or infusorial earth) from the fossil remains of microscopic diatom plants, are used to reduce gloss and increase resistance to "blackening" upon calendering. Luminosity is enhanced by fluorescent pigments having an average size of 1 micron, which improve brightness, or by phosphorescent pigments having an average size of 5-10 microns, which create an afterglow (useful for special map and display papers). Sulfides of zinc or cadmium create a short afterglow, while strontium or calcium create a long afterglow. Finally, modern coatings may contain plastic pigments such as polystyrene, which is widely used in publication-grade coated papers [12, 13, 20].

Binders

Modern coating binders provide bonding strength among the pigment particles and the rawstock. They also act as barriers controlling ink absorption and hold-out, and they influence the rheology (flow), water-holding, and set-time of wet coating. Papers with strong binding adhesion are called "hard-sized" while those with weak binding adhesion are called "soft-sized" [13, 14]. The principal binders in use today are classified as either hydrophilic water-soluble colloids based on starch (corn, tapioca, white potato, sorghum, sweet potato, rice, and wheat) and protein (glue, casein, and soy), or resin or "rubber" latexes and resin emulsions in aqueous medium, such as styrene-butadiene, polyacrylate, and polyvinyl acetate (sometimes used with starch and protein) [13, 14, 21].

Starch, because of its low cost, is the prevalent binder in the U.S. today, although to control its high viscosity and tendency to gel, retrograde, and lose strength, it must be modified by oxidation, chemical treatment (hydroxyethylation), enzyme conversion or conversion to dextrins. Oxidized starch (treated with alkaline sodium hypochlorite) is used primarily for off-machine coatings. Chemically modified starch is used for size-press coatings or trailing blade-coaters because of its high viscosity and ink receptivity. Starch converted by enzymes (inactivated by heating to 205–212 degrees F for 10–20 minutes or by phosphates, silicates, acids, formaldehyde, etc.) is the principal binder for machine-coated publication papers because of its low cost and ease of adjustments. Conversion to dextrin is done by dry-roasting starch at low temperatures for British gum, which is the best dextrin for coatings as compared to canary or white dextrin, produced by high temperatures and acid treatment. Water-resistant starch is required for coated lithographic paper, tag papers, washable wallpaper, fancy wraps, etc. Compared to casein, starch binders can be produced with less foam, odor and spoilage, although the final coating may have lower bonding strength, water resistance, finish and oil absorption.

Animal glue (bone or preferably hide) is presently used only for coated specialty papers such as playing cards, wallpapers, metal-coated papers, and grades needing high gloss and water resistance. Technical grade gelatin is used with barium sulfate for photographs, with glycerol, sorbitol, and butanol added for conditioning. Glue can be hardened for water-resistance with chrome alum or formaldehyde treatment, although it never becomes as resistant as casein. Casein is today used for off-machine coating of very high-grade printing papers and for machine-coating of paper boards. It is made from curd from coagulated skim milk, acid-separated and alkaline treated, which none-the-less retains some fat that may result in the formation of grease spots. It has a high degree of water-resistance if treated with formaldehyde, zinc salt solution, alum acids, or lime. Soybean protein from soy flour is used in washable wallpaper, and as fluid extender in casein coating mixtures, although it is not as readily waterproofed.

Resin latex or emulsion binders are used for both off- and on-machine coating (generally in combination with starch, soybean protein, or casein), to increase stabilization against mechanical breakdown from high shear on roll-coaters; to improve water-holding properties; and to reduce costs. Latexes can plasticize starch coatings for calendering, flexibility, and wet-rub resistance. Synthetic resin latexes and emulsions have good bonding strength, smoothness, flexibility,

dimensional stability, curl resistance, wet-rub resistance, and gloss, and some of these properties even improve with age. Styrene-butadiene latex, which provides a very smooth surface, high finish, and crisp edge when folded, has been used as the sole binder in many modern European and Japanese mills [23]. These resin and latex binders calender easily because of thermoplastic qualities, but they may also "blacken" or darken on calendering.

Other binders used in lesser amounts are polyvinyl alcohols which have high pigment bonding strength but also high cost, and water-soluble cellulose derivatives which have good bonding strength, flexibility and oil resistance, but high viscosity. Other water soluble resins may include polyacrylic acid, polyacrylamide, and polyacrylonitrile.

Additives

Modern additives serve many purposes and may include eveners (levelers and smoothers), pigment-dispersing agents (fluidifiers, stabilizers, softeners, plasticizers, lubricants), and anti- and defoaming agents, etc. [14]. Coatings with starch binders may require leveling agents or soaps such as sodium, ammonium or calcium stearates (which also minimize dusting and increase gloss during calendering); fluidifiers and viscosity stabilizers of urea and dicyandiamide, or polyoxyethylene stearate, polyethylene glycol laurate and sulfated oleic acid (used with enzyme-modified or thermochemically-modified starches); softening agents including invert sugar, sorbitol, urea, glycerine, or corn syrup (to increase pliability); and plasticizers or lubricants such as calcium stearate, resin latexes, ethanolamine soaps, alkyd resins, fatty acid esters, soaps, and polyglycols. Coatings with casein, soy protein, resin latex and resin emulsion binders may generate foam, which can form pinholes and craters as air bubbles burst through the coating upon drying. Foam may be composed of fine bubbles, dispersed throughout the coating mixture, or coarse froth along the top of the mixture. Antifoamers include sulfonated oils, pine oil, amyl alcohol, skim milk, ether, kerosene, tributyl phosphates, silicones, and other agents which depress surface tension and convert fine foam, which undermines coating weight and structure, into coarse froth expelled from the surface of the coating. Defoamers may be added to coating formulas to lower surface tension if foaming has already begun (0.1–1.0 % total weight of solids; too much can cause problems in printing offset papers) [14]. Latex binders might require stabilizers of anionic and nonionic surfactants such as sodium dodecyl sulfate, polyethylene oxide alkyl, or alkyl/aryl ethers.

Other additives might include smoothing agents, improving flexibility and preventing pinholes, such as pine oil (also a preserving agent) and sulfonated oils (0.1–0.5% total volume of coating mix; too much can cause oil spots). Carboxymethylcellulose can control viscosity and minimize binder migration. Amines increase gloss. Wax emulsions (paraffin or microcrystalline) decrease dusting during calendering, and increase water repellency, flexibility and gloss. Carnauba wax may be used with friction calendering for high gloss. Shellac dispersed in ammonia has been used in paper coating formulas to increase water resistance [14].

Finishing

Modern finishing may include varnishing with a clear spirit varnish to improve durability, to protect a final printed surface, and to increase luster. However, the amount of varnish gloss depends on the hold-out quality of the coating, which is enhanced by a binder having good film-forming properties (such as casein as opposed to starch) and a less absorbent pigment (such as fine clay rather than calcium carbonate).

Modern coated papers, formerly flint and friction calendered (which used talc as a finishing pigment), are today usually supercalendered (sometimes with emulsions including wax, soap, or polyethylene), which subjects the coating to pressure-polishing, improving smoothness and gloss, with the least reduction in bulk and light-scattering coefficient [14]. The level of gloss is influenced

primarily by three factors. One factor affecting gloss is the supercalendering process, which imparts different effects depending on the type of calender used (e.g. having stacks of 10–12 soft cotton-filled rollers); the number of nips through which the sheet is passed; the pressure, which may range from 400 to 2000 pounds per linear inch; the speed, which may reach 800 meters per minute or more; and the temperature, since heat increases supercalendering action by softening the web and plasticizing the binder. A second factor affecting gloss is the binder and its amount (higher ratios lower gloss), as well as type (i.e. starch produces a dull gloss while resins and latexes are glossiest, although latexes have a tendency to "blacken" from too close compression and the resultant loss of light-refracting pigment surfaces). A third factor affecting gloss is the pigment and its moisture content, which along with heat serves to soften and plasticize the coating so that supercalendering does not crush the paper structure below (although high moisture can also cause "blackening"); pigment particle shapes, such as clay's plate-like particles; and pigment particle size (the finer, the higher the gloss in general) [13,14].

Calendering lowers the strength of coated papers one wax level in the TAPPI Standard Dennison Wax Pick Surface Strength Test, and can cause unwanted patterns of ridges and valleys if coatings become over-plasticized, as with some high speed roller-coating systems. Another polishing technique is cast-coating, which can be done on or off the papermaking machine. It requires pressing a freshly coated paper against a highly polished nickel or chromium plated drier; after the paper dries, it is stripped off (wax is sometimes added to aid in stripping). The technique, low-speed and high-cost, can be used with greater amounts of binder but yields a soft porous surface, susceptible to marring [14].

Conclusion

Variables of furnish (composition, size, refractive index, brightness, and pH of pigments particles, as well as type or amount of binders and additives), and of formation (application and calendering procedures), affect the physical structure of a pigment coating, which in turn affects the optical properties of a pigment-coated paper [13, 14]. Unfortunately, the structural and optical properties of a coating may also change as a result of degradation and subsequent conservation treatment, affecting the ultimate appearance of the coating. However, there are trends in the type of furnish materials and formation procedures employed in the production of coated papers that may aid in the characterization of pigment-coated papers requiring conservation treatment. By using the information on pigment elemental composition, size, refractive index, brightness and pH in Table I, coupled with binder analysis, conservators may be able to identify the type of pigment coatings with which they are confronted. By incorporating information on use, rawstock, brightness, and gloss set by the US Grading Standards, in Table II, they may be able to identify the grade of a given pigment-coated paper. In understanding the effects of furnish and formation procedures on coating properties, and by correlating the information in Tables I and II, conservators may be able to better anticipate and evaluate changes in properties that might occur from conservation treatments.

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