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The Effect of Chemical Cross-linking Agents on
Starch and Starch- latex Adhesive Mixtures in
Coating Color Formulations

Steven F. Rehrauer

1971

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Printing Science and Engineering

ABSTRACT

Various representative coatings were prepared, run, and resulting papers evaluated with great emphasis placed on keeping conditions constant throughout all experimental work. The study was limited to two types of starches; regular and cationic and two types of insolubilizers; melamine formaldehyde and glyoxal. The effect of adding some latex to the adhesive mixture, with respect to water resistance, was also explored.

Data received substantiated the superiority of glyoxal over melamine formaldehyde in producing wet-rub resistance in starch base coatings. Calculated results also proved the merit of using a cationic starch, in conjunction with the insolubilizing additives, rather than a regular starch. However, data also showed that neither melamine formaldehyde nor glyoxal alone in a starch paste ever surpassed a starch paste with small amounts of latex added, with respect to water resistance.

Finally, representative samples of all test coatings made were printed on an offset printing press. Single-pass printing quality of all sheets, irregardless of the coating mixture, was excellent. Multi-pass printing was not explored. The water resistance of all of the samples was sufficient to produce excellent printing quality with no signs of sheet curl.

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HISTORICAL BACKGROUND

Wet calendering, offset printing, outdoor exposure, and water contact with labels represent some of the many reasons why coatings must be resistant to moisture. The rate at which it is developed and the degree required cover a wide range. Therefore, these coatings require adhesives that have a high pigment-binding capacity and suitable rheological properties. Alternation of the structure of starch by enzyme conversion and by chemical modification has given viscosity stability, high clarity, good flow, and superior water retention properties to starch pastes (6, 5).

The requirement of specific performance for binders is illustrated by the problems encountered from the trend toward lower basis-weight coatings in publication grade papers. To function well in these circumstances, an adhesive must not only contribute satisfactory rheological properties but must also impart suitable strength and water resistance at low adhesive-to-clay ratios.

Chemical reaction between the coating adhesive and the "additive" is the usual mechanism to obtain water resistance. Ideally, addition to the wet coating system is preferred, provided undesirable changes in rheology can be avoided (6).

Starch, protein, and synthetic adhesives vary in their inherent ability to resist water damage. Starch coatings can be insolubilized by blending with resins or latexes which are water resistant. The water

sensitive chemical groups of the starch adhesive can be reacted with a resin which blocks the tendency to go into solution. Another mechanism is to use cross-linking chemicals that cause the adhesive to polymerize and reduce the tendency to redisperse in water.

Starch insolubilization has been accomplished by reactions with formaldehyde which is acid catalyzed to yield water insoluble compounds. Condensation products of formaldehyde with phenols, melamine, and urea develop suitable wet-rub resistance with starch upon aging. It is thought that the aldehyde and the hydroxyl groups of the starch form a complex insoluble compound. The cross-linking of starch molecules proceeds until the starch loses its ability to swell in water (6).

It is doubtful how far urea and melamine formaldehyde resins really react with starches. Most of their action in the insolubilizations of starches consists of the covering of the starch films with insoluble condensed resin. There is, however, thought that some cross-linking with the starch is also accomplished (6).

Cross-linking, therefore, can be of a purely chemical or of a more physical nature. Chemical cross-links between starch molecules can be formed by reaction with glyoxal, in which hemi-acetals are formed which are unstable. Then, on drying, full-acetals are formed. These are stable and render the starch insoluble.

Glyoxal greatly improves the wet-rub resistance of starch-clay paper coatings. In the past, commercially available glyoxal has been noted for

its poor color in aqueous solutions and its tendency to impart color to paper. Glyoxal is now manufactured by a process that yields virtually a water-white product that gives little or no color to the paper (7).

Adding glyoxal to the finished coating color produces the lowest viscosity, while addition of the glyoxal to the hot starch cook gives the highest viscosity. Manufactures of glyoxal state that cooking glyoxal with starch gives slightly higher wet-rub resistance. ✓

It has been found that a high degree of wet-rub resistance can be obtained with as little as four percent glyoxal (based on starch concentration) by proper selection of conditions and the use of the new processed glyoxal (7).

In the publications of the manufactures of the various cross-linking agents, comparisons of different modified starches have been presented. Those comparisons show that different types of derivatives do not lead to the same water resistance.

Therefore, it can be questioned whether the differences in obtainable water resistances originate in differences in reactivity with chemical insolubilizing agents or in differences in the pigment binding capacity. It can be argued that starch with a superior binding strength in the dry state is likely to also give an improved wet-rub resistance. If this is so, the comparison of different starches with different additives should be done on the basis of approximate molecular weight of the starch. Since



finding the actual molecular weight of a starch is impossible, viscosities of the pastes have served to give relationships to molecular weight. Therefore, by holding Brookfield viscometer readings somewhat constant for each starch mixture, one can thus control the molecular weight of the starches and receive data from which more accurate conclusions about binding capacity can be made (4).

Cationic starches are known to have superior binding capabilities over conventionally known starches. It would then seem reasonable that additives used to insolubilize starches would have more of an effect on a cationic starch than on a regular starch. Prior studies with cationic starches and wet-rub resistance additives have shown that this is true. Lower levels of the additive are generally needed to produce equal amounts of water resistance than with regular starches (3).

EXPERIMENTAL DESIGN

Number 2 coating clay was the sole pigment in the coating mixture. The pigment was dispersed through the use of a laboratory size sigma-blade mixer. Pigment solids were maintained at 75%. No dispersing additives were added to the clay mixture since the clay selected was factory pre-dispersed. Each clay batch was subjected to 30 minutes of sigma-blade action to assure a uniform pigment mixture.

All starch slurries were cooked in the presence of the pigment. This helped to obtain a homogeneous mixture and to maximize the possible binding strength. The starch slurries then were heated to 190°F and maintained there for a 20 minute cooking cycle. All cross-linking agents were added to the hot cook about 10 minutes into the cooking cycle. Constant agitation throughout the cooking cycle was provided through the use of a high speed open impeller mixer. Latex addition to the coating mixture came after the mixture had cooled to below 130°F when the cooking cycle was completed.

pH was adjusted before the starch cook and readjusted after the starch cook to about 7 with sodium hydroxide. In order to assure approximate equal molecular weight of the starches, viscosities of all coatings were adjusted to read about 600 cps at 100 rpm using a Brookfield viscometer. This necessitated adjusting coating solids from a range of 42% to 50% as indicated by each individual coating mixture. Equal coating viscosities also helped produce consistent coating weights between 9 and 11

pounds.

All coatings were rod coated using a #10 rod on a laboratory Keegan Coater. Adjustments were made during each run to assure consistent coating weight and quality throughout the runs. All coated sheets were then exposed to Tappi humidity conditions for two weeks before any evaluation testing was performed. This gave coatings, using melamine formaldehyde as an insolubilizing agent, time to cure and develop wet strength resistance.

Certain standard coatings were performed from which all other test coating data was compared. Binders used in the standard coatings included:

1. A commercially used converted starch alone.
2. A cationic starch alone.
3. A commonly used latex in conjunction with the above converted starch.
4. The above latex in conjunction with the above used cationic starch.
5. The latex alone.

Each subsequent test coating used one of the first four above adhesive systems with one of the two insolubilizing agents under investigation. Varying percent additions of the chemical insolubilizers were used in order to get an idea of the amount of each agent needed to produce optimum results.

The converted starch used for testing was Penford Gum 280. The cationic starch used was Katobond 15. All latex used was Dow Latex 636. The melamine formaldehyde selected for evaluation was Scripset 101 manufactured by the Monsanto Company. Glyoxal produced by Union Carbide, Paper Chemicals Division was also tested for insolubilizing strength.

Testing procedures used were all Standard Tappi procedures except for the wet-rub test and the water resistance test. The wet-rub; finger method test was run similar to the Tappi Routine Control RC-184 testing procedure. It involves immersing samples of the coated sheets in distilled water for ten seconds, laying the samples on black paper and firmly stroking the wet surface with the forefinger five times so that any loosened coating is transferred to the black paper. After drying, the brightness of the spot on the black paper is determined. Low brightness readings indicate good wet-rub resistance. The water resistance; turbidity method test was as a modification of Tappi Routine Control RC-185 testing procedure. Since my intent here was to receive data concerning the general water resistivity of the coated papers produced, not their wet-rub resistance, abrasion to the coating surface wanted to be minimized. To do this I used a magnetic stirring bar to merely produce motion of the coated paper in the water solution; instead of a nylon bristle brush on a Taber Abraser as indicated in the Tappi procedure. Twenty one-half-inch squares

of each coating sample were separately introduced to 200 milliliters of distilled water at 25°C and allowed to mix for 15 minutes on top of a magnetic stirrer. At the end of this time the resulting water solutions were read on a Beckman B Spectrophotometer at 600_{mm} wave length using distilled water as a blank. Readings for light transmitted through the cells was recorded. Low readings indicate low water resistance. Readings approaching 1.00 indicate good water resistance.

Tabulated test data appear on the following pages.

TABLE I

OK

Standard Coating Runs

	<u>Coat Weight</u> 25 X 38 = 500(#)	<u>Brightness</u>	<u>Opacity</u>	<u>Gloss</u> 8 nips (%)	<u>K & N</u>	<u>Wet-Rub</u> <u>Finger Method</u> <u>% Brightness</u>	<u>Water Resistance</u> <u>Turbidity Method</u> <u>% Transmittance</u>
Base Stock	0	82.5	81.2	34.7	30.9	-	-
18% PG 280	11	80.5	88.3	56.4	46.6	46.8	.09
18% Cationic	10	79.7	86.9	55.8	59.9	44.6	.18
15% Latex	11	78.8	90.3	64.3	55.0	6.3	Past Full Scale
6% Latex 12% Cationic	9	79.5	87.4	59.1	61.0	26.3	.98
6% Latex 12% PG 280	9	80.2	88.8	56.5	54.6	28.4	.94

TABLE II

Ethylated Starch Coatings With Glyoxal Addition

	<u>Coat Weight</u> <u>25 X 38 = 500(#)</u>	<u>Brightness</u>	<u>Opacity</u>	<u>Gloss</u> <u>8 nips (%)</u>	<u>K & N</u>	<u>Wet-Rub</u> <u>Finger Method</u> <u>% Brightness</u>	<u>Water Resistance</u> <u>Turbidity Method</u> <u>% Transmittance</u>
18% PG 280 2% Glyoxal	9	80.2	88.8	55.3	49.9	36.7	.47
18% PG 280 5% Glyoxal	9	80.3	88.4	58.7	52.2	36.1	.49
18% PG 280 10% Glyoxal	9	80.6	88.3	54.8	53.8	36.3	.52

TABLE III

Cationic Starch Coating With Glyoxal Addition

	<u>Coat Weight</u> <u>25 X 38 - 500(#)</u>	<u>Brightness</u>	<u>Opacity</u>	<u>Gloss</u> <u>8 nips (%)</u>	<u>K & N</u>	<u>Wet-Rub</u> <u>Finger Method</u> <u>% Brightness</u>	<u>Water Resistance</u> <u>Turbidity Method</u> <u>% Transmittance</u>
18% Cationic 2% Glyoxal	9	78.9	88.1	57.8	60.6	33.9	.78
18% Cationic 5% Glyoxal	9	78.5	88.4	55.5	60.4	32.9	.82
18% Cationic 10% Glyoxal	9	77.3	87.9	56.3	59.2	33.7	.83

TABLE IV

Latex-Ethylated Starch Coatings With Glyoxal Addition

	<u>Coat Weight</u> <u>25 X 38 = 500 (#)</u>	<u>Brightness</u>	<u>Opacity</u>	<u>Gloss</u> <u>8 nips (%)</u>	<u>K & N</u>	<u>Wet-Rub</u> <u>Finger Method</u> <u>% Brightness</u>	<u>Water Resistance</u> <u>Turbidity Method</u> <u>% Transmittance</u>
3% Latex 15% PG 280 2% Glyoxal	10	79.8	89.9	60.3	55.8	33.7	.87
3% Latex 15% PG 280 5% Glyoxal	11	80.0	89.5	59.7	56.0	31.0	.91
3% Latex 15% PG 280 10% Glyoxal	9	79.7	88.9	61.6	57.0	31.3	.90

TABLE V

Latex-Cationic Starch Coatings With Glyoxal Addition

	<u>Coat Weight</u> <u>25 X 38 = 500(#)</u>	<u>Brightness</u>	<u>Opacity</u>	<u>Gloss</u> <u>8 nips (%)</u>	<u>K & N</u>	<u>Wet-Rub</u> <u>Finger Method</u> <u>% Brightness</u>	<u>Water Resistance</u> <u>Turbidity Method</u> <u>% Transmittance</u>
3% Latex 15% Cationic 2% Glyoxal	9	78.1	88.9	57.2	60.1	32.1	.90
3% Latex 15% Cationic 5% Glyoxal	11	77.2	88.7	61.4	60.8	31.9	.92
3% Latex 15% Cationic 10% Glyoxal	10	77.0	88.5	55.9	62.5	32.4	.83

TABLE VI

Ethylated Starch Coatings With Melamine Formaldehyde Addition

	<u>Coat Weight</u> <u>25 X 38 = 500(#)</u>	<u>Brightness</u>	<u>Opacity</u>	<u>Gloss</u> <u>8 nips (%)</u>	<u>K & N</u>	<u>Wet-Rub</u> <u>Finger Method</u> <u>% Brightness</u>	<u>Water Resistance</u> <u>Turbidity Method</u> <u>% Transmittance</u>
18% PG 280 5% M.F.	11	80.9	89.4	59.3	39.6	38.1	.33
18% PG 280 10% M.F.	9	81.0	89.4	61.4	39.1	37.2	.32
18% PG 280 15% M.F.	9	81.0	89.9	57.4	40.1	37.5	.35

TABLE VII

Cationic Starch Coatings With Melamine Formaldehyde Addition

	<u>Coat Weight</u> 25 X 38 = 500(#)	<u>Brightness</u>	<u>Opacity</u>	<u>Gloss</u> 8 nips (%)	<u>K & N</u>	<u>Wet-Rub</u> <u>Finger Method</u> <u>% Brightness</u>	<u>Water Resistance</u> <u>Turbidity Method</u> <u>% Transmittance</u>
18% Cationic 5% M.F.	10	79.9	88.6	55.4	58.5	34.7	.56
18% Cationic 10% M.F.	10	79.9	88.5	59.7	54.6	35.6	.52
18% Cationic 15% M.F.	10	80.2	88.2	57.7	50.8	33.4	.68

TABLE VIII

Latex-Ethylated Starch Coatings With Melamine Formaldehyde Addition

	<u>Coat Weight</u> 25 X 38 = 500(#)	<u>Brightness</u>	<u>Opacity</u>	<u>Gloss</u> 8 nips (%)	<u>K & N</u>	<u>Wet-Rub</u> <u>Finger Method</u> <u>% Brightness</u>	<u>Water Resistance</u> <u>Turbidity Method</u> <u>% Transmittance</u>
15% PG 280 3% Latex 5% M.F.	10	80.3	89.5	60.1	55.4	35.1	.88
15% PG 280 3% Latex 10% M.F.	11	80.0	89.7	59.1	50.8	34.8	.86
15% PG 280 3% Latex 15% M.F.	10	80.9	89.3	59.5	48.4	35.4	.85

TABLE IX

Latex-Cationic Starch Coatings With Melamine Formaldehyde Addition

	<u>Coat Weight</u> <u>25 X 38 = 500(#)</u>	<u>Brightness</u>	<u>Opacity</u>	<u>Gloss</u> <u>8 nips (%)</u>	<u>K & N</u>	<u>Wet-Rub</u> <u>Finger Method</u> <u>% Brightness</u>	<u>Water Resistance</u> <u>Turbidity Method</u> <u>% Transmittance</u>
15% Cationic 3% Latex 5% M.F.	11	79.5	88.5	58.4	59.5	34.6	.87
3% Latex 15% Cationic 10% M.F.	10	80.0	88.5	58.6	57.4	33.9	.89
15% Cationic 3% Latex 15% M.F.	11	79.9	89.8	57.2	54.0	34.2	.91

DISCUSSION

As was stated earlier in this paper, all test procedures were impartial and consistent for all samples. Great care was taken to assure that the test values obtained were representative of the sample involved. Better insight into the data obtained can be gained through individual discussion of the specific tests run.

Coat Weight

Coat weight was an important variable of the coatings, and had to be kept constant. Coat weight influences the results received from other test data taken. Therefore, for quantitative test results to be possible, the coat weight must be held constant. Recorded data shows that this was accomplished with a coat weight of ten pounds, plus or minus one pound, based on a 25 X 38 = 500 ream size sheet. Coat weights this close throughout all runs were possible by keeping the viscosities of all coatings close to a steady figure.

25
38

200
75

95

Brightness

In all test cases brightness readings were lower than that of the base stock. This was expected, since #2 coating clay was used which has low brightness. Brightness tests were run to evaluate the binder and hardness effects. Data collected by this investigator showed that brightness readings were pretty much unaffected by the type of coating mixture used. Variations were recorded throughout all the test coatings, but to try and draw conclusions from such slight changes would be, it seems, a futile effort. Aside from the initial drop in brightness values from

the base stock to any coated paper, any varying in the brightness values can be said to result from testing errors.

The above statements on brightness particularly substantiate statements made by the manufacturers of glyoxal that their product does not adversely affect brightness readings of coated papers. There has been some concern that glyoxal did in fact impart a color to finished coated papers and thus reduce brightness values. Testing showed this concern to be unwarranted.

Opacity

Opacity values were taken to show the relative opacifying power of the various coating mixtures. As can be expected, the opacity of a base stock was lower in all cases to that of the coated papers. This is due to the mere added thickness of the paper stock by the coating and does not necessarily reflect on the type of coatings used in my work. This is because the opacity is more a function of the pigment used than it is of the binder system selected. In my coatings the pigment used was kept constant, therefore indicating that opacity readings should also stay somewhat constant. The variations received in opacity data can be said to be a result of mainly testing discrepancies and only slightly to that of the adhesive used. It was, however, shown that none of the adhesive mixtures investigated seriously hampered development of the opacifying power present in the pigment.

Gloss

Gloss readings, again, are more a function of the type of pigment

used than they are of the binder present. Relative hardness of the binder adhesion does, however, effect gloss capacity. All sheets produced were run through eight nips of a supercalender, thus developing what could be considered the maximum gloss attainable for each coating mixture. However, gloss values varied across the board for all test samples. No visible trend was indicated for any series of coating runs. The reason for this, I think, is a result of supercalendering variables combined with gloss reading discrepancies. As with opacity readings, the only true value gained by presenting the gloss data lies in establishing that the various adhesive mixtures did not seriously hamper the development of the coatings gloss capabilities.

K & N Ink

Testing for K & N Ink absorption indicates the ease to which printing ink will penetrate into a sheet. The amount of penetration is measured by the decrease in brightness of the sheet after the test is run. Data collected hopefully simulates the sheets ink receptivity when run on a printing press.

Ink receptivity is not only a function of the pigment used in the coating, but is also definitely influenced by the adhesive character. Since the pigment type was kept constant throughout all coatings, any changes in the K & N Ink values would be a direct result of the adhesive paste employed. Data comparison indicated that all coatings in which the cationic starch was used gave higher K & N Ink values than the corresponding coatings with the regular converted starch. This leads

to the conclusion that the cationic starch used (Kato-Bond 15) is less ink reseptive than the regular converted starch tested (Penford Gum 280).

Also noted was the lower K & N Ink values for all runs subject to melamine formaldehyde addition; verses the corresponding runs with glyoxal addition. This result indicates that coatings employing melamine formaldehyde as the insolubilizing agent will produce sheets with more ink receptivity than will coatings with glyoxal added as the insolubilizing agent. Correlating the data within each coating series is on much shakier footing. There seems to be a tendency in all coating systems involving glyoxal addition for less and less ink receptivity with increased additions of glyoxal. This is shown by the increasing K & N Ink values as glyoxal addition is stepped up. The opposite holds true for all those coatings in which melamine formaldehyde is used as the insolubilizing agent. With these coating formulations, employing melamine formaldehyde as the insolubilizer, K & N Ink values decrease with increased percent addition of the wet-rub agent. This points toward increased ink receptivity with greater amounts of melamine formaldehyde in the coating color.

Wet-Rub; Finger Method

The one giant variable in the finger method test is the amount of pressure put on the sheet when stroking it with the forefinger. If rubbing pressure cannot be controlled the worth of the test is, at the least, somewhat limited. To help control this variable I taped a one kilogram weight on my forefinger and rubbed the sheets in such a fashion

as to put no other downward pressure on the sheet as my finger was dragged across it, except that of the standard weight. This rubbing system was employed on all coated sheets tested.

Despite the efforts made to reduce the operating variables of the test, data still remained somewhat scattered within each series of test coatings. With data from this test, it is therefore impossible to make accurate conclusions as to what percent addition of the cross-linking chemicals gives optimum wet-rub resistance. ✓ Certain generalities were evident, however. One was that no coating system with only three percent latex added ever recorded better wet-rub readings than either of the standard runs with six percent latex, irregardless what insolubilizing agent was added. This holds true even when maximum additions of the insolubilizing agents were added. Also the superiority of glyoxal for producing wet-rub resistance was noted. All coating series using glyoxal attained better wet-rub resistance readings than did the corresponding coating series with melamine formaldehyde.

Water Resistance; Turbidity Method

Data recorded here surprisingly corresponding quite well to the data taken for wet-rub resistance; finger method. No test coating system achieved transmittance readings as high as the standard coatings with six percent addition of latex. Also the superiority of glyoxal over melamine formaldehyde as a producer of water resistance was shown through the light transmittance data. Within each coating series, transmittance

data was also shown to scatter, thus again making it impossible to tell at what percent addition of the insolubilizing agents maximum water resistance was achieved. This is probably due to some discrepancy in the testing procedure, unknown to this investigator.

Printed sheets were not presented because there were no distinguishing factors in the print quality from any of the test coatings used. All coated sheets produced excellent printing quality irregardless of the coating system used in preparing the sheets for printing. All sheets were one-pass printed on a multilith offset press. Curl problems in the resulting sheets was also negligible. Multi-pass color printing was not done because of the lack of available equipment to do the job. It is possible that printing problems would have developed if multi-pass color printing were done on some of the coated papers produced. However, no evidence of any printing problem occurred with the one-pass printing undertaken.

CONCLUSIONS

Data taken indicates the relative merit of using any insolubilizing agent at all for one-pass offset printing as unnoticed. Truly, adding some insolubilizing agent into a coating system to produce sheets more suited for one-pass offset printing would be a fruitless venture. However, addition of the insolubilizing agent did increase the wet-rub and water resistance over the standard starch runs with no additives at all. This could help overcome possible printing problems when multi-pass color printing is needed. Glyoxal seemed to insolubilize the coatings better than melamine formaldehyde. But neither glyoxal nor melamine formaldehyde surpassed latex as an insolubilizing additive. Latex, with its inherent problems plus higher cost, still seems to be the best and most assured way to insolubilize a starch based coating.

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