Resin Bonding of Offset Papers Containing Mineral Fillers

Martin J. O’Leary, Bourdon W. Scribner, and Joshua K. Missimer

Papernaking studies at the National Bureau of Standards have shown that melamine-formaldehyde resin used as a binding agent in offset lithographic papers containing no mineral fillers has the beneficial effects of improved printing quality, decrease in the beating time of the fibers, and large increase in permissible content of hardwood fibers. The last is desirable for the conservation of the scarcer softwood fibers.

The previous work has shown that as little as 1½ hours of beating gives satisfactory results with the usual 50 percent of hardwood fibers increased to 75 percent, when 1 to 3 percent of melamine resin is added.

Further investigation has been made to determine whether these beneficial effects occur when the mineral fillers, clay and titanium dioxide, are used. With ½ hours of beating of a fiber furnish consisting of 75 percent of soda pulp (hardwood fibers) and 25 percent of sulfite pulp (softwood fibers) 15 percent of clay made the papers too soft for lithographic papers, lowering all of their desirable physical properties. The addition of the melamine resin had little or no beneficial effect on these papers. However, with a 50-percent-sulfite, 50-percent-soda, 15-percent-clay furnish, the physical properties were improved by the addition of 3 percent of melamine resin, resulting in good lithographic papers. The clay retention was lowered slightly by the addition of 3 percent of melamine resin, and more so with 1 percent of melamine resin. When the stock was given 5½ hours of beating, the melamine resin was of assistance in producing improved lithographic papers made from 75 percent of soda pulp and 25 percent of sulfite pulp.

When 3 percent of titanium dioxide was added to both 50-percent soda, 50-percent sulfite, and 75-percent soda, 25-percent sulfite furnishes with 5½ hours of beating and 1 or 3 percent of melamine resin added, satisfactory offset papers were produced. The retention of the titanium dioxide was decreased somewhat by the resin.

As reported in an earlier publication, the retention of both fillers was improved by using an excess of aluminum sulfate and adjusting the stock to the desired pH with sodium carbonate.

1. Introduction

Previous investigations in the paper mill of the National Bureau of Standards on the use of melamine-formaldehyde resin as a binding agent in offset papers, have shown that the resin imparted better printing quality, decreased the beating time required for the fibers, and permitted the use of a greatly increased proportion of hardwood fibers in the papers. As little as 1½ hours of beating gave satisfactory results with the usual 50 percent of the short, weak hardwood fibers increased to 75 percent. [1, 2] The use of the larger content of hardwood fibers is desirable for the conservation of the scarcer softwood fibers.

In the previous work, no mineral filler was used. This is a desirable component because it fills the interstices between the fibers of paper and imparts a better finish and higher opacity, all of which are favorable for improved printing quality. This article gives the results of an investigation of the influence of mineral fillers on the beneficial effects of the binding resin, and the effect of the resin on the retention of the fillers in the paper.

2. Experimental Papernaking Equipment

The papernaking equipment at the Bureau is semicommercial in size and is adapted to the experimental manufacture of papers under conditions that simulate those of typical industrial plants. Detailed description and photographs of the essential equipment are contained in previous publications [3, 4].

The equipment used in this work consisted essentially of a 50-lb beater with a copper-lined wooden tub and manganese-bronze bars and plate; a Jordan refiner with bars of bronze and steel alloy; a four-plate, flat screen and a 29-in. fourdrinier papermaking machine with a wire 33 ft in length; two presses; nine 15-in. dryers; a machine calender stack of seven rolls; a reel; a supercalender with 5 rolls; 3 steel rolls; 2 cotton rolls; and a 38-in. trimmer.

3. Papernaking Raw Materials

Different percentages of the following commercial pulps were used in this study: (1) Bleached sulfite, made by cooking Eastern spruce wood, a softwood, in a solution or calcium bisulfite. This is a relatively standard quality of pulp such as is commonly used for offset paper. (2) Bleached soda, a filler pulp produced by cooking poplar wood, a hardwood, in a strong solution of caustic soda. This is an unusually strong soda pulp for a short fibered stock.

The chemical characteristics of the pulps used are shown in table 1.

The fillers were 15 percent of clay and 3 percent of titanium dioxide. Their chemical compositions are given in table 2. The fillers were mixed with water and screened through an 80-mesh wire to remove dirt or foreign ingredients.
A acidity in which mined sulfate were 85 percent, costing a total area
A detailed description of the roll setting was
A minimum retention of the pigment is sheared to the beater, because it
A mixture that received
A = percentage of ash in oven-dry stock going to
A = percentage of ash in oven-dry paper at reel;
A one-percent rosin size precipitated with aluminum sulfate was used on all the runs.
A Melamine-formaldehyde resin 607 of the American Cyanamid Co. was used as the binding agent.
A all percentages of materials added to the pulp are based on the oven-dry (110° C) weight of the pulp.

4. Manufacturing Procedure

Fifty pounds of fibers was furnished to the beater in each instance. When clay was added, the fibers were 85 percent, and the clay was 15 percent of the furnish, making a total of 58.8 lb in the beater.

When titanium dioxide was used, 97 percent of the furnish was fibers and 3 percent titanium dioxide, making a total of 51.55 lb.

An excess of alum was used when a filler was furnished to the beater, because it was found in the study of experimental manufacture of paper for war maps [5], that by adding an excess of alum and then adjusting the pH with sodium carbonate, the retention of the pigment was increased resulting in a decided improvement in the opacity of the paper. A minimum and a medium beating time were used. A detailed description of the roll setting may be found in previous publication on offset papers [6].

With the minimum beating time, 1½ hours, the stock received a mixing just long enough to thoroughly separate the fibers and give fillers, size, and alum a good mixing. Such treatment does not develop strength in the paper.

With the medium beating time, 5½ hours, the stock received a light brushing for ½ hour. This treatment slightly frays out the fibers, causing them to interlace better in forming a sheet of paper. It also makes the surface a little harder, which helps to minimize fuzz.

A longer beating time creates a gelatinous substance on the fibers. It also makes a hard, brittle sheet of paper. The more gelatinous substance that is combined with the finished paper, the more the paper will expand when it becomes wet and contract when it dries. This expansion and contraction of the paper cause misregister in offset lithographic printing.

The beaten stock was dropped to the chest and pumped in a continuous stream through the stuff box into the jordan.

The stuff box is divided into three compartments: one section receives the stock from the pump; the second compartment has a gate that regulates the desired amount of stock delivered to the machine per hour; the third section receives the surplus stock, which then returns to the machine chest.

The jordan was used only for mixing the fibers with the same setting for all runs.

From the jordan the stock drains into a fan pump and is mixed with the white water from the paper machine. The mixture is then pumped to a riffle box that contains several baffle boards. The stock flows over these baffle boards, and the heavy particles, such as sand, metal, etc., settle to the bottom of the box. From the riffle box the stock flows through a screen with slots cut 0.018 in., thence to the headbox onto the wire.

Melamine-formaldehyde resin was added in the form of a colloidal solution, made by dissolving the powdered resin in warm water acidified with hydrochloric acid.

The formula used in this study was submitted in 1943 by the American Cyanamid Co.

39 pounds of 20° Be hydrochloric acid is added to 50 gallons of water and heated to 140° F; 100 pounds of melamine resin 607 is added slowly to the acid solution while stirring; the resin solution is added to cold water to make a total volume of 100 gallons.

The resin was added at the screen hopper, after the stock had left the screens and before the head box. There are two reasons for adding the resin at this particular place; first, melamine resin has an affinity for dirt, therefore, it should be added at a point after the dirt has been removed; second, melamine resin should have the minimum amount of agitation to obtain the maximum efficiency of wet strength in the paper, but should be added at a point where the resin will have time to obtain uniform distribution in the stock before it flows onto the wire. The temperature of the stock was maintained at 90° ± 2 deg F. One percent of rosin size was used in each of the papers. It was precipitated with papermaker's alum, Al₂(SO₄)₃, which was used to control the pH of the stock.

The finish imparted by the small machine calenders was low, therefore a light supercalendaring of the paper was necessary, so that the finish would be comparable to that of commercial papers.

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Table 1. Test data on pulp used in experimental papers

<table>
<thead>
<tr>
<th>Kind of pulp</th>
<th>Alpha cellulose</th>
<th>Beta cellulose</th>
<th>Gamma cellulose</th>
<th>Penetrons</th>
<th>Copper number</th>
<th>Ash b</th>
<th>Natural resin b</th>
<th>Acidity (class electrode), hot water extraction</th>
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</thead>
<tbody>
<tr>
<td>Sulfite</td>
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<td>11.3</td>
<td>2.5</td>
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<td>0.42</td>
<td>pH</td>
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<td>Soda</td>
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<td>1.80</td>
<td>0.58</td>
<td>6.6</td>
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</table>

* Based on total cellulose.

Table 2. Composition of fillers used

<table>
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<tr>
<th>Fillers</th>
<th>Loss at 150° C</th>
<th>Further loss on ignition</th>
<th>SiO₂</th>
<th>Fe₂O₃</th>
<th>Al₂O₃</th>
<th>TiO₂</th>
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<tr>
<td>Clay</td>
<td>%</td>
<td>%</td>
<td>%</td>
<td>%</td>
<td>%</td>
<td>%</td>
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<td>.15</td>
<td>None</td>
<td>None</td>
<td>None</td>
<td>96.2</td>
</tr>
</tbody>
</table>

Retention = 0.94B(100 - C - A) / A(100 - C - B), in which
A = percentage of ash in oven-dry stock going to machine;
B = percentage of ash in oven-dry paper at reel;
C = percentage of oven-dry filler lost on ignition.

One-percent rosin size precipitated with aluminum sulfate was used on all the runs.

Melamine-formaldehyde resin 607 of the American Cyanamid Co. was used as the binding agent.

All percentages of materials added to the pulp are based on the oven-dry (110° C) weight of the pulp.
5. Testing
All physical and chemical tests of the pulps and papers were made in accordance with the standard methods of The Technical Association of the Pulp and Paper Industry.3

The pH of mill waters at the beater, stuff box, and head box was determined with a glass electrode.

6. Interpretation and Discussion of Test Data
To enable the reader to interpret the graphs more clearly, a dividing line has been drawn through the graphs. All test results above the line are considered good and all those below the line, poor.

Some of the important tests are the pick, expansion, oil penetration, opacity, curl, tear, bursting strength, and folding endurance. It is impossible to develop a paper whose characteristics are good in all respects. In order to develop a paper with a low curl, low expansion, and a low oil penetration time, which are essential in lithographic papers, the process usually adversely affects the strength and pick. A low pick causes much trouble in lithographic printing because the printing blanket picks bunches of fibers from the paper and in a short time the blanket and plate fill up, necessitating a shutdown to clean the press. According to Robert F. Reed and James J. Spevecak [7], “Bond strength is measured by the wax pick test, and a pick of six or better is generally conceded to be necessary”. Using this statement as a base, a wax pick of 6A was placed on the line; therefore, any pick test shown above the line will be considered good, and any test shown below the line, poor.

The pick, bursting strength, and folding endurance correlate. When the pick increases, the bursting strength and folding endurance increase also, and vice versa. An average was taken of the bursting strength, tensile strength, tearing strength, and folding endurance for the six papers in table 3 having a pick of 6A, and the average values were placed on the line also. They are comparable to those of book papers when clay is used. The other values on the median line are based on the previous investigations of offset papers by the Bureau [8].

7. Results of Tests
The test data for the experimental papers are given in table 3 and shown graphically in the figures.

Figure 1 shows the results obtained with a furnish of 75 percent of strong soda pulp and 25 percent of Eastern sulfite pulp; beating time was 1½ hours. This furnish, with melamine added, was found satisfactory in the previous work. Fifteen percent of clay, an amount normally used for offset papers, softened the paper and adversely affected the desirable physical properties of folding endurance, pick, and bursting and tensile strengths. Also, the clay resulted in lint or fuzz on the wire side of the paper, the side next to the paper machine wire. The fuzz and the pick correlate—the lower the pick, the more fuzz occurs on the paper. The cause of fuzz, in most cases, is a free under-beaten stock. This gives the paper a soft surface. The greater part of the surface friction is on the wire side, and this friction causes the loosely bound fibers to fluff up on that side. The clay has the same effect on the paper in these respects as unbeaten free stock.

The addition of 3 percent of melamine resin in the absence of clay (run 1564) increased the bursting strength 100 percent, folding endurance 300 percent, tensile strength 55 percent, and the pick about 300 percent, compared to paper made without resin and clay (run 1562). However, the paper made with 15 percent of clay and 3 percent of melamine resin (run 1593) had physical properties comparable to those of the paper made without resin and clay (run 1562). Papers with such low physical properties would not be suitable for lithographic papers because of the tacky ink that is used in lithographic printing. The blanket would pick bunches of fibers from the paper, and in a short time the blanket and plate would be filled up, necessitating a shutdown to clean the press. The clay had little effect on the tear. The retention of clay was lowered considerably with 1 percent of the resin (run 1592) but only slightly when 3 percent was used (run 1593).

Figure 2 shows the results for a series of papers made with 50 percent of strong soda pulp and 50 percent of Eastern sulfite pulp with a beating time of 1½ hours. Comparing the clay-free papers (runs 1540, 1560, and 1561) with the corresponding papers made with 15 percent of clay (runs 1594, 1595, and 1596), the tests show that the clay again had a deleterious effect. However, the 3 percent of melamine resin (run 1596) strengthened the paper containing the clay to a degree that made it satisfactory for lithographic printing, as it had good pick, low expansion, low oil penetration time, low curl, and good bursting strength. The paper made with 15 percent of clay required 1 percent of melamine resin (run 1595) to bring the physical properties

3 Available from the Association headquarters at 122 East Forty-Second Street, New York 17, N. Y.
## Table 3. Comparative data on effect of melamine-formaldehyde resin on offset papers containing mineral fillers

<table>
<thead>
<tr>
<th>Run</th>
<th>Beater furnish</th>
<th>Filler</th>
<th>Melamine resin</th>
<th>Basis weight (lb/1000 ft²)</th>
<th>Thickness (mil in)</th>
<th>Bursting strength (kgf)</th>
<th>Machine</th>
<th>Cross</th>
<th>Tensile strength (kgf)</th>
<th>Machine</th>
<th>Cross</th>
<th>Tear resistance (inches)</th>
<th>Expansion in relative humidity (15% change)</th>
<th>Smoothness ( obligated )</th>
<th>Pick (Ostendorf, 40)</th>
<th>Oil penetration</th>
<th>Air permeability</th>
<th>Acidity (Butterworth)</th>
<th>Acid in stock</th>
<th>Ash in paper</th>
<th>Ash in filler</th>
<th>Retention of filler</th>
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<td>k</td>
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<td>%</td>
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<td>53.2</td>
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<td>0.07</td>
<td>0.18</td>
<td>94</td>
<td>80</td>
<td>5A</td>
<td>31</td>
<td>44</td>
<td>4.7</td>
</tr>
</tbody>
</table>

*All physical tests made under condition of 50-percent relative humidity and 73°F, by the Standard Methods of the Technical Association of the Pulp and Paper Industry.*

### Notes:
- Beater furnish for each run with clay was 85 percent fibers and 15 percent clay.
- Beater furnish for each run with TiO₂ was 97 percent fibers and 3 percent TiO₂.
The retention of clay in the paper without resin was about the same as in the preceding series. To offset the low physical properties caused by the addition of clay, fibers of the remaining runs were given the medium beating of 5½ hours with a light brushing. In runs using 15 percent of clay without resin with 50-percent sulfite, 50-percent soda and 25-percent sulfite, 75-percent soda furnishes, the 5½-hours beating did not improve the physical properties to any appreciable extent. The clay retention was increased somewhat.

Figure 3 shows a comparison of the effects of the 1½ (run 1595) and 5½ (run 1617) hours of beating time for papers made with 50 percent of sulfite pulp, 50 percent of soda pulp, 15 percent of clay, and 1 percent of melamine resin. The figure shows that even with 5½ hours beating the paper would be on the borderline for a good lithographic paper. However, the advantages of the latter paper were a 100-percent improvement in the wax pick and a minimum of fuzz. Figure 4 shows that the addition of 3 percent of melamine resin (run 1599) made a good lithographic paper with improved physical properties compared to those of papers made with 1 percent of melamine resin (run 1617) or no resin (run 1616). The retention of clay was better than that of the preceding two series, figures 1 and 2.

Figure 5 shows comparison tests of 5½ hours of beating time, 75-percent soda, 25-percent sulfite, using a filler.

Figure 6 shows the results for papers made with 25 percent of sulfite pulp, 75 percent of soda pulp, 3 percent of titanium dioxide, and 0 (run 1608), 1 (run 1609), and 3 (run 1610) percent, respectively, of melamine resin; beating time was 5½ hours. The physical properties of the resin-free paper (run 1608) were below average for a good lithographic paper, but the retention of the titanium dioxide was high. The melamine resin improved the physical properties in both runs and made these papers satisfactory lithographic papers with good folding endurance, bursting strength, tensile strength, and wax pick. The melamine resin again had an adverse effect on the retention of the filler. As the amount of resin
was increased, the retention of filler decreased inversely; however, the opacity was not affected.

Figure 7 shows the results for papers made with 50 percent of sulfite pulp, 50 percent of soda pulp, 3 percent of titanium dioxide, and 0 (run 1605), 1 (run 1620), and 3 (run 1607) percent, respectively, of melamine resin; beating time was 5½ hours. All three runs made good lithographic papers. The paper with 3 percent of melamine resin meets all the requirements of good lithographic papers, having high folding endurance, bursting strength, tensile strength, and pick number, and a low oil penetration time and curl.

8. Summary

When a beating time of 1½ hours was employed, 15 percent of clay had an adverse effect on the desirable physical properties of papers made with 75 percent of soda pulp and 25 percent of sulfite pulp; the addition of 3 percent of melamine resin did not result in the production of satisfactory papers. The clay weakened the paper, and made it softer, therefore fuzzier. The latter condition would cause trouble on the lithographic printing presses, filling the printing blanket and plate and thus necessitating frequent shutdowns to clean the press. However, when equal parts of soda and sulfite pulps were used, satisfactory papers resulted when 3 percent of melamine resin was used. The retention of clay was lowered considerably when 1 percent of melamine resin was used, but only slightly with 3 percent of the resin.

When the beating time was increased to 5½ hours, satisfactory papers were obtained from both 75-percent-soda, 25-percent-sulfite and 50-percent-soda, 50-percent-sulfite furnishes, containing 15 percent of clay and 3 percent of melamine resin. Also, the clay retention was improved somewhat.

When 3 percent of titanium dioxide was used as filler, good lithographic papers were obtained from both 75-percent-soda, 25-percent-sulfite and 50-percent-soda, 50-percent-sulfite furnishes, containing 1 and 3 percent, respectively, of melamine resin with 5½-hour beating time. The retention of the titanium dioxide decreased inversely as the melamine resin increased. The titanium dioxide improved the opacity considerably.

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9. References