SELF-SIZING OF CORRUGATING MEDIUM
Project 3321

Report One
A Progress Report
to
MEMBERS OF THE INSTITUTE OF PAPER CHEMISTRY
June 30, 1982
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A study has been made of the tendency for self-sizing (water resistance, air sizing) to develop in handsheets formed from pulps used in the production of corrugating medium. A survey of corrugating manufacturers indicated that approximately one-half of the companies contacted experience difficulties which are believed to be related to self-sized corrugating medium. Four types of pulps used in producing corrugating medium were examined in the program: a conventional neutral sulfite semichemical (NSSC) pulp, two green liquor semichemical (GLSC) pulps, an alkali-carbonate (AC) pulp, and a recycled fiber pulp. The water absorbency of handsheets formed from these pulps was determined by a waterdrop test by recording both the time required for the drop to penetrate through the sheet and the time required for the drop to be completely absorbed by the sample. The latter measurement received primary emphasis in the program. Handsheets formed from the various pulps were aged at room temperature (23°C) and at 105°C over a range in pH, i.e., 7, 9, and 11. Aging was first carried out in the absence of added chemicals and subsequently in the presence of various additives including rewetting agents, calcium chloride, and sequestering and chelating agents. Results obtained in the absence of additives showed that sheets formed from the green liquor pulps developed self-sizing at the highest rate of all samples tested; waterdrop values of 600+ seconds were obtained in all oven-aged samples within 30 minutes regardless of pH. Aging at room temperature produced similar results in one GLSC pulp and rapidly increasing water resistance in the other GLSC sample. The recycled fiber pulp was least subject to self-sizing followed by the AC pulp. The NSSC pulp occupied an
intermediate position with respect to self-sizing development. Contrary to expec-
tations, little consistency was found in the effect of pH on the development of self-sizing except in the case of the alkali carbonate pulp. The AC pulp showed the highest waterdrop values at pH 11 and the lowest at pH 7 at both aging temperatures. While the trend was consistent in that case, the order was the reverse of that expected.

Addition of anionic wetting agent to the pulp prior to sheet formation improved water absorbency to varying degrees depending upon the pulp and pH. However, the self-sizing effect was not eliminated and, in the case of one GLSC pulp, the rewetting agent provided little or no benefit. Addition of 0.1% of a nonionic surfactant at sheet forming temperatures of 23 and 60°C greatly improved water absorbency in one of the green liquor pulps, possibly to the point of accept-
ability. In general, increasing the sheet forming temperature of a GLSC pulp in the absence of surfactant reduced the self-sizing effect to the point at 90°C where self-sizing was essentially eliminated. However, since a 90°C forming temperature is an unrealistic condition, emphasis is placed on the results obtained with surfac-
tants at 23 and 60°C.

In other tests, classification of the various pulps to remove fines reduced self-sizing in all but one pulp (GLSC), which was found to retain a high level of solvent extractables and free organic acids after classification. As might be expected, the combination of classification followed by solvent extraction elimi-
nated self-sizing, but this is not a practical alternative to the surfactant treat-
ments described above.

Treatment of GLSC pulp with hydrochloric acid produced significant reduc-
tions in self-sizing, presumably due to removal of calcium and magnesium ions. The effect of added calcium chloride was found to depend on pH. Addition of the salt at
pH 7 tended to reduce self-sizing, whereas a detrimental effect was obtained at pH 11 in several cases. The use of sequestering and chelating agents produced some increases in water absorbency in GLSC pulp but, in general, the papers retained some measure of self-sizing under the conditions employed. Thus, in effect, the combination of moderately high forming temperatures and a moderate addition of wetting agent provided the most satisfactory results in this program.
INTRODUCTION

This is Progress Report One on Funded Research Project 3321 entitled "Self-Sizing of Corrugating Medium." This problem was brought to our attention by our Board of Trustees Research Advisory Committee. The problem is described briefly as one involving development of excessive sizing of corrugating medium (resistance to penetration by aqueous liquids) during normal storage of rolls of medium prior to converting in the boxboard plant. Generally no rosin or other internal sizing agent is added to the medium stock. The sizing develops from the natural fatty and resin materials in the pulp. For this reason the problem is sometimes referred to as "self-sizing" or "air-sizing." The resistance to liquid penetration is believed to be responsible for poor fluting of the medium and for inferior adhesion of liner to medium at normal corrugating speeds. Self-sizing is apparently a costly problem to many manufacturers of corrugated board.

A preliminary survey of a number of corrugators led to conflicting opinions about the nature of this problem and methods of coping with it. Some companies said that the judicious use of wetting agent controls the problem but at excessive cost for the surfactant (upward of $250,000 per year). Others said no adhesion difficulty was encountered, even with hard-sized medium, provided this fact was known before use so that suitable machine adjustments could be made. Still other companies said that self-sizing caused high-low fluting which was then responsible for poor adhesion.

These opinions indicated that further input about the problem was needed from experienced people of the industry before formulating the research plan.

A questionnaire designed to determine the nature and importance of the self-sizing of corrugating medium to the industry was mailed to 45 corrugating manufacturers. A copy of this questionnaire is included as Appendix I of this report.
Over 69% of the companies returned completed surveys. A summary of the results is presented in Table I. The responses revealed that approximately one-half of the manufacturers currently experience problems which are considered to be related to self-sized corrugating medium. Furthermore, the sizing manifests itself in more than two-thirds of the cases as an adhesion problem between the fluted medium and linerboard.

Corrugating manufacturers use a variety of remedial measures to cope with medium which becomes self-sized. The most frequent control method involves addition of a rewetting agent to the pulp prior to sheet formation. Machine processing changes (more steam and heat) during the converting operation and reduced roll inventory (less storage time) are also employed. Wood selection, pulping conditions, pulp fractionation (removal of fines), improved washing, and control of inorganic content can be optimized to reduce the tendency for self-sizing.

Economical and ecological pressures have encouraged replacement of the standard neutral sulfite semichemical (NSSC) pulping process with more alkaline pulping conditions such as GLSC and nonsulfur conditions such as AC-pulps. Also, more recycled fiber is being used. Practically no published information is available regarding how these pulping changes may affect the extent and rate of self-sizing. This information is essential for efficient control of self-sizing.

A research plan based on the survey information was formulated. The initial part of this research program was concerned with the comparative study of the self-sizing characteristics of representative NSSC, GLSC, AC, and recycled pulps. The effects of stock pH in conjunction with additions of rewetting agents were also determined. The quantity and type of organic solvent extractable material was also determined. Specifically, the project goals are:
### Summary of Survey Results

#### Occurrence of Self-Sizing

| Questionnaire Response | Yes | No | Don't
|------------------------|-----|----|------
| Currently experiencing problem? | 43 | 39 | 18
| Experienced problem in past? | 64 | 24 | 12

#### Problems Related to Self-Sizing

<table>
<thead>
<tr>
<th>Processing Variables which are Altered to Accommodate Self-Sized Mediums</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesive consumption and formulation changes</td>
</tr>
<tr>
<td>Increased energy consumption (steam &amp; heat)</td>
</tr>
<tr>
<td>Reduce machine speed</td>
</tr>
<tr>
<td>Other</td>
</tr>
</tbody>
</table>

#### Remedial Measures

<table>
<thead>
<tr>
<th>Effect of Pulp Stock</th>
</tr>
</thead>
<tbody>
<tr>
<td>Use of rewetting agents</td>
</tr>
<tr>
<td>Controlled pulping</td>
</tr>
<tr>
<td>Short inventory times</td>
</tr>
<tr>
<td>Other</td>
</tr>
</tbody>
</table>

#### Solution of Self-Sizing Problem

Should The Institute of Paper Chemistry pursue a program on self-sizing corrugating medium?

<table>
<thead>
<tr>
<th>Should</th>
<th>Yes</th>
<th>No</th>
<th>Don't</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>20</td>
<td>20</td>
<td></td>
</tr>
</tbody>
</table>
1. To evaluate the self-sizing potential of conventional NSSC, selected alkaline, and recycled fiber pulps

2. To establish economical procedures for treating the problem
EXPERIMENTAL

PULP PROCESSING AND ANALYSIS

Based on the mill survey and other contacts with industry representatives, five pulps were selected for study in the self-sizing program. These included a conventional NSSC, two GLSC, one AC, and one recycled fiber pulp.

Most of the pulps selected for study were received in a dewatered condition. The equivalent of five pounds of the dewatered pulp (dry basis) was refined in filtered tap water at approximately 3% consistency to a freeness range of 225-290 mL CSF. A Valley beater was used for this purpose. The beaten pulps were then dewatered to 18-26% consistency, and formaldehyde was added in an amount equivalent to 0.37% based on dry fiber weight. The dewatered pulp was stored at 40°F until used in subsequent analysis and/or handsheet preparations. Fiber analysis of the pulps is recorded in Table II. Chemical analysis of the pulps by emission spectroscopy is presented in Table III.

All pulps were solvent extracted to determine the amount and type of resinous materials which could contribute to self-sizing. In this direction the pulps were first washed with ethanol to remove water, followed by extraction with 2:1 benzene-alcohol in a Soxhlet extractor. The alcohol washings were combined with the benzene-alcohol extracts and the combination was reduced to dryness over steam. The composition of the extracts was determined by the classic separation procedure, and the results are recorded in Table IV.

The acidity or alkalinity of the five refined pulps and handsheets prepared therefrom was determined according to TAPPI Standard Method T 428 pm-77, and the results are presented in Table V.
<table>
<thead>
<tr>
<th>Pulp</th>
<th>Designation or Abbreviation</th>
<th>Fiber Type, %</th>
<th>Principal</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neutral sulfite semichemical</td>
<td>NSSC</td>
<td>Hardwood, 100</td>
<td>Oak</td>
<td>Beech, red gum, black gum, maple</td>
</tr>
<tr>
<td>Green liquor semichemical</td>
<td>CLSC-1 (Source 1)</td>
<td>Hardwood chemimechanical, 100</td>
<td>Red gum</td>
<td>Oak, black gum</td>
</tr>
<tr>
<td></td>
<td>CLSC-2 (Source 2)</td>
<td>Hardwood chemimechanical, 100</td>
<td>Oak</td>
<td>Yellow poplar, maple, red gum, black gum</td>
</tr>
<tr>
<td>Alkali carbonate</td>
<td>AC</td>
<td>Hardwood chemimechanical, 100</td>
<td>Populus</td>
<td>Birch, maple</td>
</tr>
<tr>
<td>Recycled container board</td>
<td>Recycled</td>
<td>Softwood kraft, 75.5</td>
<td>Hard pine</td>
<td>Douglas-fir</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Hardwood kraft, 11.0</td>
<td>Black gum, red gum</td>
<td>Maple, oak</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Hardwood NSSC, 13.5</td>
<td>Populus, red gum, black gum</td>
<td>Beech, maple</td>
</tr>
<tr>
<td>Pulp</td>
<td>Ash, %</td>
<td>B</td>
<td>Si</td>
<td>Mn</td>
</tr>
<tr>
<td>---------------</td>
<td>--------</td>
<td>----</td>
<td>----</td>
<td>----</td>
</tr>
<tr>
<td>NSSC</td>
<td>2.0</td>
<td>--</td>
<td>400</td>
<td>140</td>
</tr>
<tr>
<td>GLSC-1</td>
<td>1.6</td>
<td>2.5</td>
<td>350</td>
<td>110</td>
</tr>
<tr>
<td>GLSC-2</td>
<td>1.8</td>
<td>1.6</td>
<td>86</td>
<td>95</td>
</tr>
<tr>
<td>Alkaline carbonate</td>
<td>0.9</td>
<td>.62</td>
<td>110</td>
<td>34</td>
</tr>
<tr>
<td>Recycled</td>
<td>2.0</td>
<td>11</td>
<td>2800</td>
<td>64</td>
</tr>
<tr>
<td>Pulp</td>
<td>Total Extractives, %</td>
<td>Free Acids, %</td>
<td>Neutrals, %</td>
<td>Unsaponifiables</td>
</tr>
<tr>
<td>----------------------</td>
<td>----------------------</td>
<td>---------------</td>
<td>-------------</td>
<td>-----------------</td>
</tr>
<tr>
<td>NSSC</td>
<td>4.69</td>
<td>0.46</td>
<td>0.14</td>
<td>0.083</td>
</tr>
<tr>
<td>GLSC-1</td>
<td>5.47</td>
<td>0.68</td>
<td>0.16</td>
<td>0.099</td>
</tr>
<tr>
<td>GLSC-2</td>
<td>9.20</td>
<td>0.89</td>
<td>0.18</td>
<td>0.10</td>
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<tr>
<td>Alkali-carbonate</td>
<td>4.44</td>
<td>0.38</td>
<td>0.15</td>
<td>0.084</td>
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<tr>
<td>Recycled</td>
<td>2.78</td>
<td>0.45</td>
<td>0.63</td>
<td>0.44</td>
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<tr>
<td>Classified a</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NSSC</td>
<td>1.50</td>
<td>0.20</td>
<td>0.10</td>
<td>0.024</td>
</tr>
<tr>
<td>Classified a</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GLSC-1</td>
<td>3.55</td>
<td>0.36</td>
<td>0.08</td>
<td>0.025</td>
</tr>
<tr>
<td>Classified a</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GLSC-2</td>
<td>4.49</td>
<td>0.52</td>
<td>0.04</td>
<td>0.040</td>
</tr>
<tr>
<td>Classified a</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Alkali-carbonate</td>
<td>1.95</td>
<td>0.20</td>
<td>0.08</td>
<td>0.034</td>
</tr>
</tbody>
</table>

*Material retained on combination of sieves.*
TABLE V

ACIDITY OR ALKALINITY OF PULP AND HANDSHEET EXTRACTS

<table>
<thead>
<tr>
<th>Pulp Sample</th>
<th>pH of Pulp as Received</th>
<th>SO₃, %</th>
<th>Na₂O, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>NSSC</td>
<td>6.0</td>
<td>0.0028</td>
<td></td>
</tr>
<tr>
<td>GLSC-1</td>
<td>7.4</td>
<td>--</td>
<td>0.0098</td>
</tr>
<tr>
<td>GLSC-2</td>
<td>7.6</td>
<td>--</td>
<td>0.0096</td>
</tr>
<tr>
<td>Alkali carbonate</td>
<td>7.4</td>
<td>--</td>
<td>0.0059</td>
</tr>
<tr>
<td>Recycled</td>
<td>5.4</td>
<td>0.0112</td>
<td></td>
</tr>
</tbody>
</table>

Handsheet Sample<sup>a</sup>

| GLSC-1 | -- | -- | 0.0211 |
| GLSC-2 | -- | -- | 0.0434 |
| Alkali carbonate | -- | -- | 0.0149 |

<sup>a</sup>Prepared at pH 11.

HANDSHEET PREPARATIONS AND TESTING

All handsheets were prepared in a Rapid-Kothen mold (8-inch diameter). The required amount of dewatered pulp for a given series of handsheets (usually 25 grams o.d. basis) was dispersed at 1.25% consistency in a British disintegrator for a total of 300 counts. The pulp was then transferred to a stainless steel container where it was diluted to 0.5% consistency with room-temperature deionized water. The pH of the pulp was adjusted to the desired level, and additives, when employed, were stirred in for a minimum of five minutes before handsheet preparation. Sufficient pulp for a single handsheet was then metered into the sheet mold. Deionized water preadjusted to the same pH level as that of the pulp slurry was added, resulting in a forming consistency of 0.1%. The fiber slurry in the mold was stirred for 10 minutes...
with an agitator consisting of four paddles set at an angle of 90 degrees to each other and centered in the sheet mold about 0.25 inch from the 100-mesh stainless steel forming wire. The paddles were power driven and mechanically arranged so as to produce an oscillating motion covering a sweep of 60 degrees. As soon as the agitation was stopped, the stirrer was removed from the mold and the fiber drained immediately onto the forming wire. The handsheet weight was 3.85 g (o.d. basis), equivalent to 134 g/m². After forming, the sheets were couched from the wire onto blotter stock and then pressed for five minutes at 50 lb pressure. The sheets were dried in contact with one blotter on a steam-heated drum for seven minutes at 105°C.

Porosity and smoothness are among those properties known to affect wetting and penetration of liquids in porous media. Accordingly, porosity and smoothness measurements were made on handsheets formed from the selected pulps before and after solvent extraction or fiber classification. Results are recorded in Table VI.

In most cases water absorbency was measured on sheets aged at 23°C and 50% RH and on sheets subjected to accelerated aging.

Accelerated aging of handsheets was carried out in an oven at 105°C. Sheets were cut into strips (1-1/2 by 3 inches) stacked six deep and placed in a clean 100 by 15-mm Petri dish. The Petri dishes were cleaned in chromic acid cleaning solution followed by rinsing with tap water and finally with distilled water. The dishes were then allowed to air dry. The covered dishes containing the handsheet strips were placed in an oven at 105°C. After 30 minutes, the sample under the top strip was removed and the other five were left in the oven for further aging. Three additional strips were removed in the same manner after oven aging 1.0, 2.0, and 4.0 hours. The oven-aged strips were conditioned at 73°F and 50% RH a minimum of 30 minutes before testing for water resistance.
TABLE VI
SELECTED PROPERTIES OF HANDSHEETS PREPARED FROM VARIOUS PULPS

<table>
<thead>
<tr>
<th>Pulp Used to Prepare Handsheets</th>
<th>pH</th>
<th>Bendtsen Porosity, mL/min (wire side)</th>
<th>Bendtsen Smoothness, mL/min</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Wire</td>
<td>Felt</td>
</tr>
<tr>
<td>NSSC</td>
<td>7</td>
<td>74</td>
<td>3373+</td>
</tr>
<tr>
<td>NSSC</td>
<td>11</td>
<td>137</td>
<td>3373+</td>
</tr>
<tr>
<td>GLSC-1</td>
<td>7</td>
<td>186</td>
<td>3373+</td>
</tr>
<tr>
<td>GLSC-1</td>
<td>11</td>
<td>235</td>
<td>3373+</td>
</tr>
<tr>
<td>GLSC-2</td>
<td>7</td>
<td>&lt;18</td>
<td>3373+</td>
</tr>
<tr>
<td>GLSC-2</td>
<td>11</td>
<td>&lt;18</td>
<td>3373+</td>
</tr>
<tr>
<td>Alkali-carbonate</td>
<td>7</td>
<td>283</td>
<td>3180</td>
</tr>
<tr>
<td>Alkali-carbonate</td>
<td>11</td>
<td>268</td>
<td>3220</td>
</tr>
<tr>
<td>Recycled</td>
<td>7</td>
<td>129</td>
<td>2913</td>
</tr>
<tr>
<td>Recycled</td>
<td>11</td>
<td>82</td>
<td>3016</td>
</tr>
<tr>
<td>Extracted NSSC&lt;sup&gt;a&lt;/sup&gt;</td>
<td>7</td>
<td>117</td>
<td>3373+</td>
</tr>
<tr>
<td>Extracted GLSC-1&lt;sup&gt;a&lt;/sup&gt;</td>
<td>7</td>
<td>496</td>
<td>3373+</td>
</tr>
<tr>
<td>Extracted GLSC-2&lt;sup&gt;a&lt;/sup&gt;</td>
<td>7</td>
<td>&lt;18</td>
<td>3373+</td>
</tr>
<tr>
<td>Extracted alkali-carbonate&lt;sup&gt;a&lt;/sup&gt;</td>
<td>7</td>
<td>236</td>
<td>3268+</td>
</tr>
<tr>
<td>Extracted recycled&lt;sup&gt;a&lt;/sup&gt;</td>
<td>7</td>
<td>135</td>
<td>2805</td>
</tr>
<tr>
<td>Classified NSSC&lt;sup&gt;b&lt;/sup&gt;</td>
<td>7</td>
<td>3373+</td>
<td>3373+</td>
</tr>
<tr>
<td>Classified GLSC-1&lt;sup&gt;b&lt;/sup&gt;</td>
<td>11</td>
<td>3373+</td>
<td>3373+</td>
</tr>
<tr>
<td>Classified GLSC-2&lt;sup&gt;b&lt;/sup&gt;</td>
<td>11</td>
<td>3340</td>
<td>3373+</td>
</tr>
<tr>
<td>Classified alkali-carbonate&lt;sup&gt;b&lt;/sup&gt;</td>
<td>11</td>
<td>3373+</td>
<td>3271</td>
</tr>
</tbody>
</table>

<sup>a</sup>Handsheets were prepared from benzene-alcohol extracted pulps.
<sup>b</sup>Classified pulp was that portion retained on combination of sieves.

The degree of water resistance or self-sizing was determined simultaneously by two methods, i.e., through penetration and waterdrop time. For this purpose a 50-microliter drop of room-temperature distilled water was placed on the surface of
the handsheet. Through penetration was determined by measuring the time required for the water to soak through the sheet and appear on the underside. A mirror positioned at an angle under the sheet was used in determining through penetration. The time required for the drop to completely soak into the sheet was recorded as the water-drop time. Emphasis in this report is placed on the waterdrop values.

Sizing values for the NSSC pulp at pH levels of 7, 9, and 11 are presented in Table VII and Fig. 1. Comparable data for the GLSC-1, GLSC-2, alkali-carbonate, and recycled fiber pulps are recorded in Tables VIII-XI and Fig. 2-4. Note: No figure is included for the GLSC-2 pulp, since the waterdrop values were 600+ seconds without aging.

**TABLE VII**

**WATER ABSORBENCY IN HANDSHEETS PREPARED FROM CONVENTIONAL NSSC PULP**

<table>
<thead>
<tr>
<th>Aging Time, pH</th>
<th>Through Penetration, sec</th>
<th>Through Waterdrop, sec</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wire Felt Av.</td>
<td>Wire Felt Av.</td>
</tr>
<tr>
<td>23°C</td>
<td>105°C</td>
<td></td>
</tr>
<tr>
<td>7.0 0.5</td>
<td>4 3 4 169 133 151</td>
<td>21 19 20 600+ 466 533+</td>
</tr>
<tr>
<td>7.0 1.0</td>
<td>4 3 4 165 157 161</td>
<td>45 47 46 600+ 600+ 600+</td>
</tr>
<tr>
<td>7.0 2.0</td>
<td>3 2 3 141 127 134</td>
<td>600+ 458 529+ 600+ 600+ 600+</td>
</tr>
<tr>
<td>7.0 4.0</td>
<td>3 3 3 185 156 171</td>
<td>600+ 600+ 600+ 600+ 600+ 600+</td>
</tr>
<tr>
<td>9.0 0.5</td>
<td>3 2 3 229 147 188</td>
<td>600+ 600+ 600+ 600+ 600+ 600+</td>
</tr>
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<td>600+ 600+ 600+ 600+ 600+ 600+</td>
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<td>10 11 11 163 176 170</td>
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<tr>
<td>11.0 1.0</td>
<td>3 3 3 200 172 186</td>
<td>15 11 13 242 198 219</td>
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<td>11.0 2.0</td>
<td>3 2 3 211 145 178</td>
<td>18 13 16 405 287 346</td>
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<td>11.0 4.0</td>
<td>3 2 3 188 155 172</td>
<td>17 14 16 513 369 441</td>
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</table>
Figure 1. The effect of aging on waterdrop absorbency of handsheets prepared from NSSC pulp.
### TABLE VIII

WATER ABSORBENCY IN HANDSHEETS PREPARED FROM GREEN LIQUOR SEMICHEMICAL PULP (GLSC-1)

<table>
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<th>Aging Time, pH hr</th>
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<th>Waterdrop, sec</th>
<th>Through Penetration, sec</th>
<th>Waterdrop, sec</th>
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<td>Av.</td>
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<td>1</td>
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<td>1</td>
<td>1</td>
<td>600+</td>
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<td>340</td>
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<td>2</td>
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<td>600+</td>
</tr>
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<td>19</td>
<td>600+</td>
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<td>23</td>
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<td>25</td>
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### TABLE IX

WATER ABSORBENCY IN HANDSHEETS PREPARED FROM GREEN LIQUOR SEMICHEMICAL PULP (GLSC-2)

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<th>Aging Time, pH hr</th>
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<th>Waterdrop, sec</th>
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<td>Felt</td>
<td>Av.</td>
<td>Wire</td>
</tr>
<tr>
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<td></td>
<td></td>
</tr>
<tr>
<td>23°C</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7 0.5</td>
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<td>20</td>
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<td>37</td>
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<tr>
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<td>600+</td>
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<td>600+</td>
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</table>
### TABLE X

WATER ABSORBENCY IN HANDSHEETS PREPARED FROM ALKALI-CARBONATE PULP

<table>
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<th>Aging Time (pH hr)</th>
<th>Through Penetration (sec)</th>
<th>Waterdrop (sec)</th>
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<td>Wire Felt Av.</td>
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<td></td>
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<tr>
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<td>2 2 2 35 33 34</td>
</tr>
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<td></td>
<td>2.0</td>
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<td>4.0</td>
<td>2 2 2 49 48 49</td>
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<tr>
<td>105°C</td>
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<td></td>
</tr>
<tr>
<td>7</td>
<td>0.5</td>
<td>3 2 3 80 39 60</td>
</tr>
<tr>
<td></td>
<td>1.0</td>
<td>11 9 10 198 163 181</td>
</tr>
<tr>
<td></td>
<td>2.0</td>
<td>28 30 29 600+ 600+ 600+</td>
</tr>
<tr>
<td></td>
<td>4.0</td>
<td>135 72 104 600+ 600+ 600+</td>
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</tbody>
</table>

### TABLE XI

WATER ABSORBENCY IN HANDSHEETS PREPARED FROM RECYCLED FIBER PULP

<table>
<thead>
<tr>
<th>Aging Time (pH hr)</th>
<th>Through Penetration (sec)</th>
<th>Waterdrop (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wire Felt Av.</td>
<td>Wire Felt Av.</td>
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<tr>
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<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
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<td>2 2 2 93 70 82</td>
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<td></td>
<td>2.0</td>
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<td>4.0</td>
<td>2 2 2 103 105 104</td>
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<tr>
<td>105°C</td>
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<td></td>
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<tr>
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<td>0.5</td>
<td>5 5 5 249 167 208</td>
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<td></td>
<td>1.0</td>
<td>10 10 10 381 304 343</td>
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<tr>
<td></td>
<td>2.0</td>
<td>21 18 20 600+ 600+ 600+</td>
</tr>
<tr>
<td></td>
<td>4.0</td>
<td>41 47 44 600+ 600+ 600+</td>
</tr>
</tbody>
</table>
Figure 2. The effect of aging on waterdrop absorbency of handsheets prepared from GLSC-1 pulp.
Figure 3. The effect of aging on waterdrop absorbency of handsheets prepared from alkali-carbonate pulp.
Figure 4. The effect of aging on waterdrop absorbency of handsheets prepared from recycled fiber pulp.
In subsequent tests, the effects on water absorbency of a commercially accepted anionic rewetting agent were examined over a range in addition level. The surfactant was added to the pulp as a 1% solution in distilled water. Addition was made at 0.5% fiber consistency prior to the sheet-forming operation. Water absorbency tests on the resulting handsheets are presented in Tables XII-XV and in Fig. 5-7.

Bauer-McNett classification data for the pulps used in this study are recorded in Table XVI, and the degree of self-sizing developed in handsheets prepared from the classified pulps is presented in Table XVII. Table XVIII shows the effect of combining classification and solvent extraction on sheet absorbency.

The hot water-extracted pulps produced in determining acidity or alkalinity were subsequently used to prepare handsheets, and the size tests are presented in Table XIX. The effects of classification, classification + benzene-alcohol extraction (B/A), and hot water extraction are summarized in Fig. 8-11.

The effects of extended room-temperature aging without and with rewetting agent are presented in Tables XX and XXI, respectively. Table XXII shows the effect on water absorbency of adding calcium chloride to the fiber suspension prior to sheet formation.

Having established that the GLSC pulps were notably more troublesome than the AC and recycled fiber pulps, the decision was made to explore means of treating GLSC pulp to eliminate or reduce its tendency to become self-sized. In this direction, GLSC-1 pulp was treated with acid and several other agents including Calgon (sodium hexametaphosphate), Versene (tetra-sodium salt of ethylenediaminetetracetic acid), and mixtures of Calgon and the anionic rewetting agent. The effect of pulp temperature at the time of sheet formation was also examined. For purposes of comparison, reference is made to the aging results for the untreated pulp previously presented in Table VIII.
<table>
<thead>
<tr>
<th>Rewetting Agent, %</th>
<th>Aging Time, hr</th>
<th>Through Penetration, sec</th>
<th>Waterdrop, sec</th>
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<td></td>
<td>4.0</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>0.025</td>
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</tr>
<tr>
<td></td>
<td>2.0</td>
<td>--</td>
<td>--</td>
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<tr>
<td></td>
<td>4.0</td>
<td>1</td>
<td>1</td>
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<td>0.125</td>
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<td>--</td>
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23°C

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<th>Through Penetration, sec</th>
<th>Waterdrop, sec</th>
</tr>
</thead>
<tbody>
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<td>Felt</td>
<td>Av.</td>
<td>Wire</td>
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<td>600+</td>
<td>600+</td>
<td>600+</td>
</tr>
<tr>
<td>600+</td>
<td>600+</td>
<td>600+</td>
<td>600+</td>
</tr>
<tr>
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<td>600+</td>
<td>600+</td>
<td>600+</td>
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<tr>
<td>600+</td>
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</tr>
<tr>
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<td>600+</td>
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<td>600+</td>
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</tbody>
</table>

105°C
## TABLE XIII

WATER ABSORPTION IN HAND SHEETS PREPARED FROM NSSC PULP 
INCORPORATING AN ANIONIC REWETTING AGENT AT PH 11 

<table>
<thead>
<tr>
<th>Time, hr</th>
<th>Penetration, sec</th>
<th>Waterdrop, sec</th>
<th>Wire Felt Av.</th>
<th>Penetration, sec</th>
<th>Waterdrop, sec</th>
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<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
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<td>1.0</td>
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<td>1.0</td>
<td>1.0</td>
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</table>
### Table XIV

**Water Absorbency in Handsheets Prepared from GLSC-1 Pulp Incorporating an Anionic Rewetting Agent at pH 11**

<table>
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<th>23°C Through Penetration, sec</th>
<th>Waterdrop, sec</th>
<th>105°C Through Penetration, sec</th>
<th>Waterdrop, sec</th>
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<td>22</td>
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</table>
TABLE XV

WATER ABSORBENCY IN HANDSHEETS PREPARED FROM ALKALI-CARBONATE AND GLSC-2 PULPS INCORPORATING AN ANIONICREWETTING AGENT

<table>
<thead>
<tr>
<th>Pulp Used</th>
<th>Rewetting Agent, %</th>
<th>pH</th>
<th>Through Penetration, sec</th>
<th>Waterdrop, sec</th>
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<td></td>
<td>Wire</td>
<td>Felt</td>
</tr>
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<td>Alkali-carbonate</td>
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<td>0.025</td>
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<td>1</td>
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<td></td>
<td>0.05</td>
<td>11</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>0.125</td>
<td>11</td>
<td>1</td>
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<td>17</td>
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<td>12</td>
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<td>7</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>0.125</td>
<td>7</td>
<td>6</td>
<td>5</td>
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<td>600+</td>
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</table>

NOTE: All samples aged 1/2 hour at room temperature.
Figure 5. Effect of rewetting agent on waterdrop absorbency of handsheets prepared from NSSC pulp.
Figure 6. Effect of rewetting agent on waterdrop absorbency of handsheets prepared from GLSC-1 pulp at pH 11.
Figure 7. Effects of wetting agent on waterdrop absorbency of handsheets prepared from alkali-carbonate and GLSC-2 pulps.
<table>
<thead>
<tr>
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**WATER ABSORBENCY HANDSHEETS PREPARED FROM CLASSIFIED AND SOLVENT EXTRACTED PULPS**

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**NOTE:** Handsheets from the NSSC pulp were prepared at pH 7; all others at pH 11.
Figure 8. The effect of various treatments on the waterdrop absorbency of NSSC handsheets - pH 7.
Figure 9. The effect of various treatments on the waterdrop absorbency of GLSC-1 handsheets - pH 11.
Figure 10. The effect of various treatments on the waterdrop absorbency of GLSC-2 handsheets - pH 11.
Figure 11. The effect of various treatments on the waterdrop absorbency of AC handsheets - pH 11.
### Table XX

**Extended Room Temperature Aging of Handsheets**

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### TABLE XXI

**EXTENDED ROOM TEMPERATURE AGING OF HANDSHEETS CONTAINING REWETTING AGENT**

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<tr>
<td>11 0.125</td>
<td>32 22 27</td>
<td>-- -- --</td>
<td>19 29 24</td>
<td>20 28 24</td>
</tr>
</tbody>
</table>
Table XXII

The effect of calcium chloride\(^a\) on the water absorbency of handsheets

<table>
<thead>
<tr>
<th>Pulp Used</th>
<th>pH</th>
<th>Through Penetration, sec</th>
<th>Waterdrop, sec</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Wire</td>
<td>Felt</td>
</tr>
<tr>
<td>NSSC control</td>
<td>7</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>NSSC with CaCl(_2)</td>
<td>7</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>NSSC control</td>
<td>11</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>NSSC with CaCl(_2)</td>
<td>11</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Alkali carbonate control</td>
<td>7</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Alkali carbonate with CaCl(_2)</td>
<td>7</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Alkali carbonate control</td>
<td>11</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Alkali carbonate with CaCl(_2)</td>
<td>11</td>
<td>7</td>
<td>8</td>
</tr>
<tr>
<td>GLSC-2 control</td>
<td>11</td>
<td>600+</td>
<td>600+</td>
</tr>
<tr>
<td>GLSC-2 with CaCl(_2)</td>
<td>11</td>
<td>600+</td>
<td>600+</td>
</tr>
<tr>
<td>Recycled control</td>
<td>7</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Recycled with CaCl(_2)</td>
<td>7</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Recycled control</td>
<td>11</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Recycled with CaCl(_2)</td>
<td>11</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

\(^a\)100 ppm of CaO added as CaCl\(_2\).
Sheets were aged 4 hours at 23°C before measurements were made.

Acid treatments were intended to remove the adverse effects of magnesium and calcium ions on paper wettability. For this purpose, 100-g batches of the refined GLSC pulp were treated with HCl in a stainless steel container. Each batch of pulp was diluted to 20 liters with deionized water, and sufficient HCl was added to adjust the pH to 1.1. After being stirred slowly for one hour, the pulp was drained on filter paper. The pulp was then resuspended and filtered several times to remove the acid. Handsheets were prepared from this pulp at pH levels of 7, 9, and 11, and the sizing results are presented in Table XXIII.
The effect of 1, 5, and 10% Calgon on self-sizing in handsheets prepared from GLSC pulp was examined. Calgon was added as a 1% solution to 0.5% consistency pulp at pH 7. It was allowed to stir with the pulp for a minimum of five minutes before handsheets were prepared. The sizing values are listed in Table XXIV.

The effect of four hours contact time at 1 and 10% addition levels of Calgon to the pulp at pH 7 was also examined. Handsheets were prepared five minutes after addition of Calgon, and then another set was prepared four hours later in the same manner as previously described. Sizing results are given in Table XXV.

Four sets of handsheets were prepared to determine the effect of a combination of 1% Calgon and 0.05, 0.075, 0.10, and 0.125% of anionic rewetting agent on the wettability of handsheets. Calgon was added to the pH 7 pulp before the surfactant and each was stirred in a minimum of five minutes before preparation of handsheets. The size test results are given in Table XXVI.
<table>
<thead>
<tr>
<th>Calgon Added, %</th>
<th>Aging Time, hr</th>
<th>Through Penetration, sec</th>
<th>Wire Felt Av.</th>
<th>Through Penetration, sec</th>
<th>Wire Felt Av.</th>
<th>Through Penetration, sec</th>
<th>Wire Felt Av.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5</td>
<td>0.5</td>
<td>3</td>
<td>192</td>
<td>3</td>
<td>190</td>
<td>3</td>
<td>189</td>
</tr>
<tr>
<td>1.0</td>
<td>0.5</td>
<td>3</td>
<td>189</td>
<td>3</td>
<td>189</td>
<td>3</td>
<td>191</td>
</tr>
<tr>
<td>2.0</td>
<td>0.5</td>
<td>3</td>
<td>223</td>
<td>3</td>
<td>232</td>
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<td>0.5</td>
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<td>256</td>
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<td>240</td>
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<td>293</td>
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<td>1.0</td>
<td>3</td>
<td>335</td>
<td>3</td>
<td>285</td>
<td>3</td>
<td>3</td>
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<td>1.0</td>
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<td>375</td>
<td>3</td>
<td>298</td>
<td>3</td>
<td>7</td>
</tr>
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<td>1.0</td>
<td>3</td>
<td>307</td>
<td>3</td>
<td>230</td>
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<td>12</td>
</tr>
<tr>
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<td>1.0</td>
<td>3</td>
<td>387</td>
<td>3</td>
<td>311</td>
<td>3</td>
<td>34</td>
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<td>0.5</td>
<td>2.0</td>
<td>3</td>
<td>225</td>
<td>3</td>
<td>199</td>
<td>3</td>
<td>184</td>
</tr>
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<td>1.0</td>
<td>2.0</td>
<td>3</td>
<td>213</td>
<td>3</td>
<td>188</td>
<td>3</td>
<td>201</td>
</tr>
<tr>
<td>2.0</td>
<td>2.0</td>
<td>3</td>
<td>265</td>
<td>3</td>
<td>191</td>
<td>3</td>
<td>223</td>
</tr>
<tr>
<td>4.0</td>
<td>2.0</td>
<td>3</td>
<td>265</td>
<td>3</td>
<td>226</td>
<td>3</td>
<td>246</td>
</tr>
</tbody>
</table>

**NOTE:** Handsheets were prepared at pH 7.
**TABLE XXV.**

**THE EFFECT OF EXTENDED CONTACT TIME OF CALGON ON THE WATER ABSORBENCY OF GLSC-1 HANDSHEETS**

<table>
<thead>
<tr>
<th>Calgon Added, %</th>
<th>Aging Time, hr</th>
<th>Through Penetration, sec</th>
<th>Waterdrop, sec</th>
<th>Through Penetration, sec</th>
<th>Waterdrop, sec</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Wire</td>
<td>Felt</td>
<td>Av.</td>
<td>Wire</td>
</tr>
<tr>
<td>1.0</td>
<td>0.5</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>217</td>
</tr>
<tr>
<td></td>
<td>1.0</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>232</td>
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<tr>
<td>1.0</td>
<td>0.5</td>
<td>2</td>
<td>1</td>
<td>2</td>
<td>112</td>
</tr>
<tr>
<td></td>
<td>1.0</td>
<td>2</td>
<td>1</td>
<td>2</td>
<td>136</td>
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<td>2.0</td>
<td>2</td>
<td>1</td>
<td>2</td>
<td>119</td>
</tr>
<tr>
<td></td>
<td>4.0</td>
<td>2</td>
<td>1</td>
<td>2</td>
<td>122</td>
</tr>
<tr>
<td>10.0</td>
<td>0.5</td>
<td>5</td>
<td>3</td>
<td>4</td>
<td>449</td>
</tr>
<tr>
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<td>1.0</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>441</td>
</tr>
<tr>
<td></td>
<td>2.0</td>
<td>4</td>
<td>3</td>
<td>4</td>
<td>427</td>
</tr>
<tr>
<td></td>
<td>4.0</td>
<td>4</td>
<td>3</td>
<td>4</td>
<td>446</td>
</tr>
<tr>
<td>10.0a</td>
<td>0.5</td>
<td>3</td>
<td>2</td>
<td>3</td>
<td>287</td>
</tr>
<tr>
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<td>1.0</td>
<td>3</td>
<td>2</td>
<td>3</td>
<td>292</td>
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<td>2.0</td>
<td>3</td>
<td>2</td>
<td>3</td>
<td>300</td>
</tr>
<tr>
<td></td>
<td>4.0</td>
<td>3</td>
<td>2</td>
<td>3</td>
<td>313</td>
</tr>
</tbody>
</table>

*Calgon stirred in furnish four hours before handsheets were prepared.

**NOTE:** Handsheets were prepared at pH 7.
TABLE XXVI
THE EFFECT OF CALGON PLUS ANIONIC REWETTING AGENT ON THE WATER
ABSORBENCY OF GLSC-1 HANDSHEETS

<table>
<thead>
<tr>
<th>Additive</th>
<th>Aging Time, hr</th>
<th>23°C Through</th>
<th>105°C Through</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Penetration, sec</td>
<td>Waterdrop, sec</td>
<td>Penetration, sec</td>
</tr>
<tr>
<td></td>
<td>Wire Felt Av.</td>
<td>Wire Felt Av.</td>
<td>Wire Felt Av.</td>
</tr>
<tr>
<td>1.0% Calgon + 0.5</td>
<td>1 1 1 1</td>
<td>48 38 43</td>
<td>1 1 1 1</td>
</tr>
<tr>
<td>0.050% rewetting agent</td>
<td>1 1 1 1</td>
<td>47 47 47</td>
<td>2 2 2 2</td>
</tr>
<tr>
<td>2.0</td>
<td>1 1 1 1</td>
<td>53 40 47</td>
<td>4 4 4 4</td>
</tr>
<tr>
<td>4.0</td>
<td>1 1 1 1</td>
<td>50 38 44</td>
<td>11 7 9 9</td>
</tr>
<tr>
<td>1.0% Calgon + 0.5</td>
<td>1 1 1 1</td>
<td>30 21 26</td>
<td>1 1 1 1</td>
</tr>
<tr>
<td>0.075% rewetting agent</td>
<td>1 1 1 1</td>
<td>34 31 33</td>
<td>1 1 1 1</td>
</tr>
<tr>
<td>2.0</td>
<td>1 1 1 1</td>
<td>42 35 39</td>
<td>1 1 1 1</td>
</tr>
<tr>
<td>4.0</td>
<td>1 1 1 1</td>
<td>41 37 39</td>
<td>7 8 8 8</td>
</tr>
<tr>
<td>1.0% Calgon + 0.5</td>
<td>1 1 1 1</td>
<td>27 20 24</td>
<td>1 1 1 1</td>
</tr>
<tr>
<td>0.1% rewetting agent</td>
<td>1 1 1 1</td>
<td>38 38 38</td>
<td>1 1 1 1</td>
</tr>
<tr>
<td>2.0</td>
<td>1 1 1 1</td>
<td>35 32 34</td>
<td>1 1 1 1</td>
</tr>
<tr>
<td>4.0</td>
<td>1 1 1 1</td>
<td>33 34 34</td>
<td>4 4 4 4</td>
</tr>
<tr>
<td>1.0% Calgon + 0.5</td>
<td>1 1 1 1</td>
<td>27 28 28</td>
<td>1 1 1 1</td>
</tr>
<tr>
<td>0.125% rewetting agent</td>
<td>1 1 1 1</td>
<td>36 35 36</td>
<td>1 1 1 1</td>
</tr>
<tr>
<td>2.0</td>
<td>1 1 1 1</td>
<td>36 31 34</td>
<td>2 2 2 2</td>
</tr>
<tr>
<td>4.0</td>
<td>1 1 1 1</td>
<td>39 37 38</td>
<td>4 4 4 4</td>
</tr>
</tbody>
</table>

NOTE: Handsheets were prepared at pH 7.
The effect of Versene 100 on the wettability of GLSC handsheets was also
examined. In this series of experiments, handsheets were prepared at pH 5 and 9
incorporating 0.4-10.0% of Versene 100. The Versene was added as a 10% solution
to the 0.5% pulp slurry. Sizing results are listed in Table XXVII.

The final series of handsheets was prepared to determine the effect of pulp
temperature at the time of handsheet formation on the wettability of the handsheets.
Handsheets were prepared over a temperature range that may be found in commercial
papermaking and somewhat above. The temperatures chosen were 7, 23, 60, and 90°C.
Handsheets were prepared at pH 7 with and without the addition of 0.1% of the
anionic and two nonionic rewetting agents. For each set of sheets, the 0.5% pulp in
the stainless steel container and the dilution water were maintained at the same
temperature. Sizing results are recorded in Table XXVIII.

As a follow-up to this work, the solvent extractables content of the
handsheets formed in the above temperature study (in the absence of rewetting
agents) was determined by extraction with 2:1 benzene-alcohol. The results are
recorded in Table XXIX.
## TABLE XXVII

THE EFFECT OF VERSENE 100 ON THE WATER ABSORBENCY OF GLSC-1 HANDSHEETS

<table>
<thead>
<tr>
<th>Aging Temperature</th>
<th>23°C</th>
<th>105°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Through</td>
<td>Waterdrop, sec</td>
</tr>
<tr>
<td></td>
<td>Penetration, sec</td>
<td>Wire</td>
</tr>
<tr>
<td>Versene 100 Added, %</td>
<td>pH</td>
<td>Aging Time, hr</td>
</tr>
<tr>
<td>0.4</td>
<td>5</td>
<td>0.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.0</td>
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<td></td>
<td></td>
<td>4.0</td>
</tr>
<tr>
<td>0.4</td>
<td>9</td>
<td>0.5</td>
</tr>
<tr>
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</tr>
<tr>
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<td></td>
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<td></td>
<td>4.0</td>
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<td>0.8</td>
<td>5</td>
<td>0.5</td>
</tr>
<tr>
<td></td>
<td></td>
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</tr>
<tr>
<td></td>
<td></td>
<td>2.0</td>
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<td></td>
<td></td>
<td>4.0</td>
</tr>
<tr>
<td>0.8a</td>
<td>5</td>
<td>0.5</td>
</tr>
<tr>
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<td></td>
<td>1.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4.0</td>
</tr>
<tr>
<td>10.0b</td>
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<td>0.5</td>
</tr>
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<td></td>
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<td>4.0</td>
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<tr>
<td>10.0ab</td>
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<tr>
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<tr>
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<td>2.0</td>
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<tr>
<td></td>
<td></td>
<td>4.0</td>
</tr>
</tbody>
</table>

---

*a* Versene stirred in furnish four hours before handsheets were prepared.

*b* Versene present in dilution water.
### TABLE XXVIII

**The Effect of Variations In Furnish Temperature on the Water Absorbency of GlSc HandSheets**

<table>
<thead>
<tr>
<th>Temp. °C</th>
<th>Additive</th>
<th>Aging Time, hr</th>
<th>Through</th>
<th>Waterdrop, sec</th>
<th>Penetration, sec</th>
<th>Through</th>
<th>Waterdrop, sec</th>
<th>Penetration, sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>23</td>
<td>-</td>
<td>0.5</td>
<td>Wire</td>
<td>127</td>
<td>131</td>
<td>132</td>
<td>129</td>
<td>247</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.0</td>
<td>Felt</td>
<td>127</td>
<td>131</td>
<td>132</td>
<td>129</td>
<td>247</td>
</tr>
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<td></td>
<td>2.0</td>
<td>Av.</td>
<td>127</td>
<td>131</td>
<td>132</td>
<td>129</td>
<td>247</td>
</tr>
<tr>
<td></td>
<td></td>
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<td>142</td>
<td>142</td>
<td>142</td>
<td>132</td>
<td>129</td>
<td>247</td>
</tr>
<tr>
<td>60</td>
<td>4%</td>
<td>1.0</td>
<td>55</td>
<td>55</td>
<td>55</td>
<td>55</td>
<td>55</td>
<td>55</td>
</tr>
<tr>
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<td>17</td>
<td>17</td>
<td>17</td>
<td>17</td>
<td>17</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4.0</td>
<td>14</td>
<td>14</td>
<td>14</td>
<td>14</td>
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<td>14</td>
</tr>
<tr>
<td>7</td>
<td>0.1% Anionic</td>
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<td>19</td>
<td>19</td>
<td>19</td>
<td>19</td>
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</tr>
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<td>4.0</td>
<td>17</td>
<td>17</td>
<td>17</td>
<td>17</td>
<td>17</td>
<td>17</td>
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<td>0.1% Anionic</td>
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<td>22</td>
<td>22</td>
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<td>26</td>
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<td>4.0</td>
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<td>33</td>
<td>33</td>
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<td>0.1% Anionic</td>
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</tr>
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<td>34</td>
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<td>34</td>
<td>34</td>
</tr>
<tr>
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**NOTE:** Handsheets were prepared at pH 7.
### TABLE XXIX

**BENZENE-ALCOHOL EXTRACTIVES OF GLSC-1 HANDSHEETS PREPARED AT VARIOUS TEMPERATURES**

<table>
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<th>Temperature of Handsheet Preparation, °C</th>
<th>Total Extractives, %</th>
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**NOTE:** Handsheets were prepared at pH 7. The extractions were carried out for 8 hours.
DISCUSSION

Results obtained in the pulp processing and analysis portion of the program (Tables II-VI) revealed several noteworthy effects. The metal (inorganic) composition of the pulps differed quite markedly (Table III). Of particular interest to self-sizing are the high calcium and magnesium levels in the green liquor pulps. The conventional NSSC pulp also had a high calcium level, but this was coupled with a high sodium content. The recycled fiber pulp contained high levels of silicon, aluminum, iron, calcium, and titanium. Most of these components were present in the secondary fiber before repulping and may not be bound in the same manner as in the virgin pulps. In general, secondary fiber pulps do not present serious self-sizing problems if the organic binders are removed in the pulping and cleaning operations.

In general, high levels of magnesium and calcium can lead to the formation of insoluble salts with the natural and fatty resin acids present in the pulp, depending on the pH. In contrast, high levels of sodium would be expected to reduce or minimize self-sizing at high pH through the formation of water-soluble soaps. These soaps, if present at sufficiently high concentrations, would form micelles which could aid in removing unsaponifiable resinous components during the washing cycles.

With respect to the organic solvent extractables (Table IV), the green liquor pulps contained high levels of extractables before and after classification, suggesting that a considerable amount of natural resins remained with the whole fibers. The green liquor pulps also contained somewhat higher levels of free acids, whereas the recycled pulp contained relatively high levels of neutrals and unsaponifiables. The latter may be due in part to the retention of adhesive components in the repulping operation. Swanson and Cordingly (1) showed that stearic acid
volatilizes at room temperature from the crystalline state and becomes adsorbed on cellulose surfaces. In the presence of calcium and magnesium ions, insoluble salts of fatty acids may form. These salts provide a more stable sizing condition, since overturning of the fatty molecules is reduced or prevented.

Handsheets formed from the various pulps had rough surfaces before and after solvent extraction or fiber classification (Table VI). Greater differences existed in porosity where, as would be expected, removal of the fines fraction (classification) greatly increased porosity. Aside from this effect, GLSC-2 showed the lowest porosity and the alkali carbonate pulp the highest porosity. Porosity is an important parameter in the penetration of liquids into porous media, as indicated by the Washburn equation:

\[
\frac{dl}{dt} = \frac{Y_L \cos \theta}{4nL}
\]  

where \( \frac{dl}{dt} \) is the rate of penetration, \( Y_L \) is the surface tension of the liquid, \( r \) is the pore radius, \( n \) is the viscosity of the liquid, and \( \theta \) is the contact angle formed by the liquid on the solid surface. Equation (1) is for an idealized system based on uniform cylindrical pores.

The effect of surface roughness is indicated by the modified Young's equation:

\[
\cos \theta_{AA} = \frac{(Y_S - Y_{SL})}{Y_L} \sigma = \sigma \cos \theta_A
\]  

where \( \theta_{AA} \) is the apparent advancing contact angle, \( Y_S \) is the surface tension of the solid, \( Y_{SL} \) is the interfacial tension, \( \theta_A \) is the real advancing contact angle, and \( \sigma \)
actual surface area is Wenzel's roughness factor, which is equal to the geometric surface area. Hence, roughness may either increase or decrease wetting and penetration depending upon whether the contact angle is acute or obtuse.

Water absorbency in sheets formed from conventional NSSC pulp (Table VII, Fig. 1) showed a marked increase in self-sizing as a function of aging time, particularly among the oven-aged samples at pH levels of 7 and 9, whereas relatively minor increases were evident at 23°C. A consistent pattern with respect to pH was not evident under the two aging temperatures.

Comparable data for the green liquor pulps are presented in Tables VIII and IX. These pulps rapidly developed a high level of self-sizing. In fact, waterdrop values of 600+ seconds were obtained in all oven-aged samples, regardless of pH. Aging at room temperature produced similar results in GLSC-2 and rapidly increased sizing in GLSC-1, as shown in Fig. 2. The rapid development of water resistance was not expected at pH 11, in which case the presence of water-soluble soaps would normally assist wetting and penetration.

Table X and Fig. 3 show the effects of aging on the development of self-sizing in the AC pulp. In this case, waterdrop values reached 600+ seconds after two hours of aging at 105°C, regardless of pH, but aging at room temperature had little effect on water absorbency. The effect of pH was consistent at both aging temperatures but, once again, the pH 11 condition proved most adverse to water absorptivity (Fig. 3).

Results obtained with the recycled fiber pulp (Table XI, Fig. 4) showed a rather slow increase in water resistance as a function of oven aging, and the levels attained were well below those in the other pulps, regardless of pH. Aging at room
temperature produced low sizing values roughly comparable to those of the AC pulp. As in the previous case, waterdrop levels remained essentially unchanged as a function of room-temperature aging time. As previously indicated, recycled fiber pulps would not be expected to produce high levels of self-sizing, since most residual resinous material is removed in the repulping operation.

The effects of adding a commercial anionic rewetting agent to the NSSC, GLSC, and AC pulp are shown in Tables XII to XV. The effects of the rewetting agent under highly sized (adverse) conditions are shown in Fig. 5 and 6. Addition of 0.05-0.125% rewetting agent to the conventional NSSC pulp provided some advantage at room temperature, particularly at pH 7 (Table XII). Similar effects were obtained at 0.125% addition after aging at 105°C. Less favorable results were obtained at pH 11 after oven aging (Table XIII). Results in Fig. 5 show the advantage for rewetting agent addition in excess of 0.05%. Some advantage is also indicated at pH 11 over pH 7 at room temperature for all surfactant additions.

The rewetting agent produced improved water absorbency in the GLSC-1 pulp at 23°C at all addition levels and at additions greater than 0.05% at 105°C (Table XIV). However, it is evident in Fig. 6 that surfactant additions greater than 0.1% would be required for high water absorbency.

Results in Table XV and Fig. 7 show that addition of the rewetting agent to the AC pulp improved water absorbency somewhat but had no effect on GLSC-2 at pH 7 and excessively high additions would be required at pH 11.

Classification of the various pulps (Tables XVI and XVII) reduced self-sizing to some extent in all pulps except GLSC-2 (compare results in Fig. 8-11). This is tentatively assumed due to the fact that GLSC-2 contained the highest levels
of solvent extractables, free acids, and unsaponifiables after classification. The combination of classification and solvent extraction essentially eliminated all evidence of self-sizing (Table XVIII). This, of course, satisfies scientific curiosity but does not represent a viable solution to the self-sizing problem.

Hot water extraction (Table XIX) greatly reduced self-sizing in GLSC-1 and the AC pulps but was less effective in the GLSC-2 pulp. Reasons for the difference in the effectiveness of hot water extraction between GLSC-1 and GLSC-2 are not apparent, but it is assumed to be related to the total solvent extractables listed in Table IV.

Extended room temperature aging (Table XX) produced high levels of self-sizing in the NSSC pulp at pH 9, but to a lesser extent at pH 7 and 11. The GLSC pulps were previously found to become rapidly self-sized, so extended aging did not produce any changes. The AC and recycled fiber pulps showed little change with extended room-temperature aging, both remaining at low sizing levels.

Results obtained in aging handsheets for extended periods in the presence of the anionic wetting agent are presented in Table XXI. The presence of rewetting agent proved to be of little or no benefit in the NSSC and AC pulps, but some advantage was obtained with the GLSC pulps at pH 11, particularly in the case of GLSC-1.

The influence of calcium chloride on water absorption is presented in Table XXII. The effect of the calcium salt depended on pH. Addition of the salt at pH 7 tended to reduce self-sizing, whereas a detrimental effect is indicated at pH 11, most notably in the AC pulp. These results are somewhat puzzling, since formation of troublesome insoluble calcium salts of the natural resinous components would be expected to occur in the pH range of 6-8 rather than at pH 11.
The remainder of the program was directed to alleviating or eliminating the development of self-sizing in GLSC pulp, which was indicated in the preceding sections to be the most troublesome. GLSC-1 was selected for this work primarily on the basis of its extremely high calcium content. The water absorption properties of this pulp are presented in Table VIII, and comparisons may be made with these results in much of the work that follows.

Treatment of the GLSC pulp with acid (HCl) prior to sheet formation produced very significant reductions in self-sizing, presumably through removal of calcium and magnesium ions (Table XXIII). This was supported by ash contents of 1.44% for untreated pulp compared with 0.19% for the acid-treated pulp.

The effectiveness of sequestering and chelating agents in reducing self-sizing by tying up or removing calcium ions is presented in Tables XXIV-XXVII. The presence of Calgon reduced the self-sizing effect in room temperature aged sheets at the 1% Calgon addition level, but little or no additional benefit was derived from 5 and 10% Calgon additions (Table XXIV). Increasing the contact time of Calgon with the pulp to four hours (Table XXV) produced little benefit in water absorptivity at the 1% Calgon level. More substantial reductions in sizing were obtained at the 10% Calgon addition level, but the waterdrop numbers remained relatively high and the addition level was excessive. Combinations of 1% Calgon and 0.5-0.125% addition of the anionic rewetting agent were examined in the next series of tests (Table XXVI). These results show that the combination of 1% of Calgon plus 0.1 or 0.125% of rewetting agent was moderately effective in reducing self-sizing although, once again, waterdrop numbers remained relatively high after oven aging. It would
appear, however, that combination of 1% Calgon plus rewetting agent was more effective than Calgon alone or extended contact with Calgon.

The effectiveness of Versene as a chelating agent is shown in Table XXVII. Where direct comparison with previous data can be made (pH 9), it would appear that 0.4% of Versene 100 produced some improvements in water absorptivity at 23°C, but little advantage was evident after oven aging. Addition of 0.8% of Versene at pH 5 effected further reductions in waterdrop values, but once again the papers retained a significant level of sizing.

The final series of handsheets examined the effect of pulp temperature during sheet formation at pH 7 in the absence and presence of the previously used anionic rewetting agent and two nonionics (Table XXVIII). While the trends in water absorbency are not necessarily consistent, the results indicate that increasing the forming temperature to 90°C in the absence of surfactant essentially eliminated self-sizing. This, of course, is an unrealistic temperature, although temperatures of this magnitude are reached in the deinking of nongroundwood broke, in which case the pulp is washed and cleaned after the cooking stage. Actually, the results for the sheets formed at 90°C in Table XXVIII compare favorably with those formed at pH 11 from the hot water extracted pulp (Table XIX). However, emphasis is placed on the lower, more practical temperatures. For example, 0.1% of the nonionic surfactants produced substantially improved water absorbency at 23 and 60°C, and these are among the most encouraging results obtained in this study. Conceivably, slightly higher levels of the surfactant or longer contact time would further improve water absorption.

The final table (Table XXIX) lists the solvent extractables content of the handsheets prepared in the controlled temperature experiments in the absence of
rewetting agent. The extractables were essentially constant at the first three forming temperatures but then declined 0.8 or 0.9% at the highest temperature, under which condition self-sizing was essentially absent. It would be desirable to have available chemical analysis of the components removed at the 60 and 90-degree treatments, since that information may provide the answer to the self-sizing problem in green liquor pulps and possibly in other pulps as well.
CONCLUSIONS

The following conclusions are drawn on the basis of the evidence provided in this study:

1. Self-sizing develops to some degree as a function of aging in the four major types of pulp used in the production of corrugating medium, although to a very minor degree in recycled fiber pulp. The order of increasing sizing (reduced water absorbency) among the pulps tested was as follows:
   a. recycled fiber
   b. AC
   c. conventional NSSC
   d. green liquor

2. With the exception of the AC pulp, pH levels in the range of 7-11 had little or no consistent effect on self-sizing.

3. Rewetting agents were more effective in reducing self-sizing in some pulps than in others. The recycled fiber pulp did not require a rewetting agent, and the green liquor pulps proved most resistant to the beneficial effects of an anionic surfactant.

4. Classification (fines removal) reduced self-sizing in most pulps but not in one GLSC.
5. Addition of sequestering or chelating agents to GLSC pulp improved water absorbency to some extent but did not eliminate self-sizing.

6. Hot water extraction or high temperature forming greatly reduced self-sizing in GLSC pulp.

7. Forming sheets at 90°C essentially eliminated self-sizing in GLSC pulp as did the combination of classification and solvent extractions.

8. The most practical solution to the self-sizing problem appears to be the addition of 0.1% rewetting agent coupled with moderately high temperature forming conditions.
ACKNOWLEDGMENTS:

The authors are indebted to Dr. Lynden J. Stryker for his contributions to this program.
LITERATURE CITED


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Senior Scientist
## APPENDIX I

### CORRUGATING MEDIUM SELF-SIZING QUESTIONNAIRE

1. Company

2. Plant location

3. Is your plant experiencing a corrugating medium problem considered to be directly or indirectly related to self-sizing?
   - Yes
   - No
   - Don't know

4. Has your plant experienced a problem with self-sized medium in the past?
   - Yes
   - No
   - Don't know

5. Have you correlated the self-sized medium with the type of pulp stock?
   - Yes
   - No
   - Don't know
   If so, check which type(s):
   - a. Conventional semichemical pulps
   - b. Green liquor pulps
   - c. Recycled fiber pulps
   - d. Other (please specify)

6. How does/did the problem using self-sized corrugating medium manifest itself?
   - Please check where appropriate.
   - a. Poor fluting (highs and lows)
   - b. Poor adhesion at the single facer
   - Green bond strength _____ Final bond strength _____
   - c. Poor adhesion at the double backer
   - Green bond strength _____ Final bond strength _____
   - d. Other (please specify)

7. What corrugating variables are altered to accommodate self-sized medium?
   - Please check where appropriate.
   - a. Reduced corrugating machine speed
   - b. Preheating the medium
   - Lower temperature _____ Higher temperature _____
   - c. Steam shower pressures (temperature)
   - Reduced pressure _____ Increased pressure _____
   - d. Adhesive consumption
   - Increased _____ Decreased _____
   - e. Change of adhesive formulation _____ How?
   - f. Increased pressure roll pressure _____ Decreased pressure _____
   - g. Other (please specify)
8. What tests are regularly conducted on the corrugating medium to predetermine if self-sizing will cause problems in the converting process?
   a. Water drop tests
   b. Other liquid penetration tests (please specify)
   c. Adhesion tests (please specify)
   d. Other (please specify)

9. Please indicate the acceptable range of values for tests in Item 8.
   | Test       | Acceptable Range | Sample Age at Test |

10. What remedial measures are taken to prevent self-sizing?
    a. Modification of the pulping process (specify)
    b. Addition of rewetting agents:
        Anionic ______ Nonionic ______ Cationic ______
    c. Web cooled after driers
    d. Reduced stock inventory times
    e. Other (please specify)

11. What are the approximate costs of the remedial treatments in terms of dollars/year for all company operations?

12. Should the Institute pursue solution or control measures of the corrugating medium self-sizing problems? Yes _____ No _____ Don't know _____

13. Comments:

   ___________________________________________________________
   ___________________________________________________________
   ___________________________________________________________

   Signature __________________________ Title _______________ Date ______

Please return the completed questionnaire in the postpaid envelope to:

SURFACE & COLLOID SCIENCE CENTER
THE INSTITUTE OF PAPER CHEMISTRY
P.O. Box 1039
Appleton, WI 54912