Repulping Bottle Carriers

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REPULPING BOTTLE CARRIERS

By

Jennifer A. Giver

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A Thesis submitted in partial fulfillment
of the course requirements for the
Degree of Bachelor of Science
Department of Paper and Printing
Science and Engineering

Western Michigan University
Kalamazoo, Michigan
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The primary object of this thesis project was to repulp post consumer bottle carriers with the addition of chemical reagents and mechanical shear. Recycling bottle carriers is difficult to achieve due to the chemical additives that are added to the pulp. Chemical additives introduced to the virgin pulp are wet strength resins.

Post consumer bottle carriers used in this experiment contained Kymene 557, which is a wet strength product produced by Hercules Incorporated and is difficult to repulp due to the cross-linking employed by this chemical additive. This experiment utilized both oxidizing and reducing chemical reagents such as hypochlorite and sodium hydrosulfite. The mechanical shear used for this experiment was from the use of the Waring Blender.

The results of this experiment showed that soaking time and temperature proved to be significant variables in this experiment. Also, the use of chemical reagents increased the easiness of breaking down the pulp. Conclusively, hypochlorite proved to be the most effective reagent in comparison to sodium hydrosulfite for yield percentage, tensile strength, and cost.
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I. INTRODUCTION

Much of today's paperboard, such as beverage carriers, that contain wet strength resins are disposed of in landfills. In fact, approximately 650,000 tons of beverage carriers are used in the United States each year, most of which are disposed of in landfills. The increasing trends to recycle and divert as much material as possible from landfills, which are being filled and closed rapidly, has led to the need to examine the possibility of reclaiming the fibrous material.

Beverage carriers are a source of paperboard that contain polymeric amine/amide-epichlorohydrin resins, that for the most part are currently landfilled. They are landfilled due to the difficulty in breaking down the wet strength resin network within the paperboard and the high cost associated with the methods being used. As the competition for secondary fibers grow, the paper industry is forced to look at resources that have been currently neglected.

This experiment concentrated on studying the effects of temperature, soaking time and blending time on repulping beverage carriers. As well, the effects of oxidizing and reducing reagents such as hypochlorite and sodium hydrosulfite were examined at various levels of concentrations. The significance of using these reagents was that hypochlorite is a lignin removing agent and sodium hydrosulfite is a lignin preserving agent. Conclusively, the pulp runs were tested for yield percentage, tensile strength and cost.
II. BACKGROUND

Increasing interest of the public for using "environmentally friendly" products has increased greatly within the last fifteen years. The paper industry has been a major contributor in helping to recycle paper. Unfortunately, wet-strength paperboard contributes a large amount of unrecoverable secondary fiber, much of which is disposed of in landfills. In fact, approximately 650,000 tons of bottle carriers are used in the United States, most of which are disposed of in landfills (1). The problem with repulping wet-strength paperboard is its resistance to breaking down when exposed to moisture. Much of this wet-strength resistance results from chemical additives in the paperboard, which provide resistance to moisture through cross-linking. This inability of repulping wet-strength paperboard is a major and continuing problem for the pulp and paper industry (2).

When water and cellulose are exposed to an aqueous media, the fiber-to-fiber bonds do not stay together and are destroyed. The addition of wet strength resins provide cross-linking between the fibers that enable the product to remain together during wet conditions. Cross-linking from the resins provide an extra bond between the fines and fibers that are not destroyed by the presence of water (3). The major disadvantage of using wet strength resins is that they are useless without the presence of hemicelluloses.
Thus only the pre-existing bonds interact with the resins, without creating any new resin bonds in the fibers (4). In order for a paper to be considered wet strength paper, it must maintain more than fifteen percent of the tensile strength when it is exposed to moisture (3).

The most common wet strength resin used in many of the paper or paperboard is a neutral/alkaline curing resin. See Figure 1(4).

**PAE CROSS-LINKING REACTION SCHEME**

![PAE Cross-Linking Reaction Scheme](attachment:image.png)

Figure 1. Neutral/Alkaline Curing Polymeric Amine/Amide-Epichlorohydrin
The resin that is used from this group is polymeric amine/amide-epichlorohydrin and the categorization of this resin is due to the backbone polymer chemistry or by the reactivity. These resins are of polyamine (amine-polyamide) and epichlorohydrin cationic water soluble condensates. They do not need acidic conditions to polymerize in the paper. Polymeric amine/amide-epichlorohydrin resins are the most important commercial thermosetting products used in producing wet strength paper. They also have the ability to be absorbed by the fiber in the neutral to alkaline furnishes (4).

Polymeric amine/amide-epichlorohydrin resins are formed by reacting an amine-containing polymer or polyamine with an epoxide that has a second functional group in a water solution. The compound that is typically used is epichlorohydrin. This epichlorohydrin alkylates and cross-links with the polyamine to a moderate molecular weight. This allows the formation of tertiary or quaternary groups which allows a cationic resin to be contained with the reactive groups. This process results in and promotes cross-linking. This reaction is then halted by reducing the pH or by dilution to produce acid salts form the converted amine groups (5). Polymeric amine/amide-epichlorohydrin resins maintain a reel wet strength of around fifty percent and after a time of approximately three weeks full wet strength properties are obtained (4).

An experiment conducted by Gruntfest and Young (5) shows that wet strength resins do not modify the fibers themselves, but only influence the fiber
bonding. The last step involved in a series of steps that create the adsorption of wet strength resins on pulp show that the resin must be at the surface of the fiber in order for to be freely adsorbed on the fiber. Studies by Epsy and Wave (6) show that polymeric amine/amide-epichlorohydrin resins are predominately self cross-linkers. They show that the cross-linking employed is from reducing the amount of excess water and thus producing swelling in the pulp (5).

Polymeric amine/amide-epichlorohydrin resins require to be thermosett and allowed a curing time period once off the reel. The first half of the wet strength is set on the paper machine. The next quarter develops during the first few days off the reel and the last quarter requires approximately three weeks. Not only is curing time required with the use of polymeric amine/amide-epichlorohydrin resins, but also the conditions of the environment during production must be strictly controlled. The factors that need to be evaluated are pH, mineral contents such as salts and alum, hardness, temperature, anionic contaminants, fines, debris, refining degree, addition point, presence of dyes and chlorine (5).

Repulping of wet-strength is accomplished by mechanical and chemical methods. The most common approach to treat wet-strength paperboard is by using chlorine-based or persulfate based compounds along with the use of intense mechanical shear. Unfortunately, the application of the intense shearing process destroys strength properties of the paperboard by
considerably reducing the fiber length. Repulping wet-strength paperboard also requires more electrical energy, thermal energy, time and money compared to using virgin pulp (2).

The significance in finding new methods for recycling wet-strength paper is beneficial for not only environmental purposes, but for marketability as well. The use of less energy, chemicals, and virgin pulp will help save our natural resources and help to preserve the natural environment.
III. PROBLEM STATEMENT

This thesis was conducted in order to determine optimum conditions for repulping bottle carrier paperboard with the use of chemical reagents and mechanical shear. An oxidizing chemical, hypochlorite, and a reducing chemical, sodium hydrosulfite, were used with the Waring Blender.

OBJECTIVES

I. The first objective was to determine the temperature, soaking time, and blending time to be used during the experiment.

II. The second objective was to do experimentation with hypochlorite and sodium hydrosulfite at different levels of concentrations.

III. The third objective was to determine from the experimental trials the pulp that provided optimum percent yield and tensile strength.
IV. EXPERIMENTAL DESIGN

This experiment was composed of two phases. The purpose of the first phase was to prepare a control run. Phase II experiments were used to determine optimum variable levels for hypochlorite and sodium hydrosulfite in blending.

Phase I

The control run involved performing preliminary experiments prior to the main experiments to determine optimum soaking time, temperature, and blending times. As well, previous works aided in determining approximate variable levels to use.

To prepare stock for each trial, 107.5 grams, at 7% moisture, of the beverage carrier paperboard was torn into small pieces that were approximately 3 X 3 squares. The bottle carrier paperboard pieces were transferred to a metal bucket on a heating plate of which individual runs were conducted at various soaking times and temperatures. When optimum levels were determined, the bottle carrier paperboard pieces were transferred to the Waring Blender where blending times of 7 and 15 minutes were run. The pulp
samples were stored at 4 C for further pulp testing of percent yield, tensile strength, and cost.

**Phase II**

Phase II involved the use of hypochlorite and sodium hydrosulfite during blending of where the blending time was determined in the control run. Hypochlorite was used at three various concentrations on 100 grams of the O.D. fiber weight to determine which concentration provides the highest experimental values of percent yield and tensile strength. The three concentrations were prepared by using 12 grams, 18 grams, and 24 grams of hypochlorite in 1000 ml graduated cylinder. The remainder of the cylinder was filled with distilled water. Thus, the percent concentration of hypochlorite used on the pulp was at 5.2%, 7.8%, and 10.4%. This experimental setup is illustrated in Table 1.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Hypochlorite Concentrations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Best Blending Time</td>
<td>5.2%</td>
</tr>
</tbody>
</table>

Sodium Hydrosulfite was used at two concentrations of low and high during blending. The two concentrations of sodium hydrosulfite were
prepared by filling a 1000 ml graduated cylinder to 26 milliliters for the first
ccentration and then again to 52 milliliters for the second concentration.
The remainder of the graduated cylinder was then filled with distilled water.
Thus, the percent concentration of sodium hydrosulfite used on 100 grams of
the O.D. fiber weight was 2.6 % and 5.2%. This experimental setup is illustrated
in Table 2.

Table 2

<table>
<thead>
<tr>
<th>Sodium Hydrosulfite Concentrations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Best Blending Time</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>2.6%</td>
</tr>
<tr>
<td>5.2%</td>
</tr>
</tbody>
</table>

After each run the pulp was stored at 4 C to await further pulp testing of
percent yield, tensile strength, and cost.

Experimental Materials

The beverage carriers were obtained from the BFI corporation. Sodium
hydrosulfite was obtained from Hoechst Chemical Company. Hypochlorite was
purchased from D&W Food Center as household bleach.
Pulp Testing

Testing involved running the pulp samples through the six cut screen to determine the percent yield. Percent yield was recorded and the pulp that went through the 6/1000" screen was used for making hand sheets on the Noble and Wood Handsheet Maker.

The samples were transferred from the six cut screen in the recycle laboratory to the wet laboratory where a slurry of 3 - 5 % was produced. The slurry was placed in the proportionator and handsheets were made in the range of 60 g/m². From each pulp sample, 10 handsheets were produced and placed in the standard conditioning room for at least three days, as recommended by TAPPI standards. After this, the sheets were ready for testing and were tested for tensile strength according to TAPPI standards. Appendix I references TAPPI standards.
V. RESULTS AND DISCUSSION

This chapter is broken down into two sections. The first section presents and analyzes data from the first phase, and the second section deals with the analysis of the data from the second phase.

Phase I

The control run was necessary to determine optimum soaking time, temperature and blending time. Some previous works aided in determining approximate variable levels to use. It was determined to soak each batch of pulp for two hours at 180 F. These conditions provided saturation of moisture to the center of the pieces of bottle carrier paperboard, whereas, less temperature and time left the pieces dry in the center. This was done by a trial and error method and it was important for the pieces to be fully saturated before blending.

Graphs were made of the data obtained to demonstrate the outcome of the results. See Table 3.

Table 3

<table>
<thead>
<tr>
<th></th>
<th>Percent Yield</th>
<th>Tensile Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control I (7min)</td>
<td>85%</td>
<td>0.6440 kN/m</td>
</tr>
<tr>
<td>Control II (15 min)</td>
<td>90%</td>
<td>0.7010 kN/m</td>
</tr>
</tbody>
</table>
When comparing the two base runs for tensile strength, control II, which was blended for 15 minutes, had a tensile strength of .7010 kN/m compared to control I, which was blended for 7 minutes, had a tensile strength of .6440 kN/m. The yield percentage was calculated and compared for this experiment. The yield that resulted from control II, which was blended for 15 minutes, resulted with 90% yield, whereas control I, which was blended for 7 minutes, resulted in 85% yield. This demonstrates two things. First, that increased blending time broke the pieces apart better and gave more usable fiber for making handsheets. Second, temperature and soaking time of 180 F for two hours assisted in penetration of the water and heat to the bottle carrier paperboard pieces before blending. Comparing the results for percent yield and tensile on the base run, the blending time of 15 minutes for control II gave better results over control I. For the majority of the discussion the base run that will be used for comparison will be control II. See Graph 1 and 2.
GRAPH 1: TENSILE STRENGTH
CONTROL I VS CONTROL II

TENSILE STRENGTH

kN/m

0.0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8

Control I (7 min)  Control II (15 min)
GRAPH 2: PERCENTAGE YIELD
CONTROL I VS CONTROL II

YIELD
%

90%
89%
88%
87%
86%
85%
84%

Control I (7 min)
Control II (15 min)
Phase II

Tensile Strength

Comparing control II with the hypochlorite runs, the hypochlorite shows a tremendous increase in tensile strength. This would result from the hypochlorite penetrating the agglomerated fibers so they would be free for exposure to the blending. The tensile strength of the hypochlorite runs varied with the amount of hypochlorite used. See Table 4.

<table>
<thead>
<tr>
<th></th>
<th>Tensile Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control II (15 min)</td>
<td>0.7010 kN/m</td>
</tr>
<tr>
<td>Hypochlorite 5.2%</td>
<td>1.0677 kN/m</td>
</tr>
<tr>
<td>Hypochlorite 7.8%</td>
<td>0.9284 kN/m</td>
</tr>
<tr>
<td>Hypochlorite 10.4%</td>
<td>0.7864 kN/m</td>
</tr>
</tbody>
</table>

The highest tensile strength of 1.0677 kN/m resulted from the hypochlorite concentration of 5.2% (on the O.D. fiber). The lowest tensile strength of 0.7864 kN/m resulted from the hypochlorite concentration of 10.4%. The lowest value is still considerably higher than the value from control II. The optimum value of the three concentrations tested with hypochlorite was at 5.2%. See Graph 3.
The use of sodium hydrosulfite versus the control II still proved to be
generally more effective. See Table 5.

<table>
<thead>
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<th></th>
<th>Tensile Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control II (15 min)</td>
<td>.7010 kN/m</td>
</tr>
<tr>
<td>Sodium Hydrosulfite 2.6%</td>
<td>.8326 kN/m</td>
</tr>
<tr>
<td>Sodium Hydrosulfite 5.2%</td>
<td>.7758 kN/m</td>
</tr>
</tbody>
</table>

The highest tensile strength value of the sodium hydrosulfite concentration of
2.6% was .8326 kN/m and the lowest tensile strength value of the
concentration of 5.2% was .7758 kN/m. The values are still higher than not
using chemical reagents, but not as effective for producing a tensile strength of
1.0677 kN/m as from the 5.2% concentration of hypochlorite. See Graph 4 and
5.
GRAPH 3: TENSILE STRENGTH
CONTROL II VS HYPOCHLORITE

- Control II (15 min)
- Hypochlorite 5.2%
- Hypochlorite 7.8%
- Hypochlorite 10.4%
GRAPH 4: TENSILE STRENGTH
CONTROL II VS SODIUM HYDROSULFITE

Control II (15 min) | Sodium Hydrosulfite 2.6% | Sodium Hydrosulfite 5.2%
Graph 5: Tensile Strength
Sodium Hydrosulfite vs Hypochlorite
Percent Yield

Comparing control II with the hypochlorite runs, the hypochlorite runs had moderate yield percentage. See Table 6.

Table 6

<table>
<thead>
<tr>
<th>Percent Yield</th>
<th>Control II (15 min)</th>
<th>Hypochlorite 5.2%</th>
<th>Hypochlorite 7.8%</th>
<th>Hypochlorite 10.4%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>90%</td>
<td>63%</td>
<td>67%</td>
<td>72%</td>
</tr>
</tbody>
</table>

The lowest percentage yield of 63% resulted from the concentration of 5.2%. The highest yield percentage of 72% resulted from the concentration of 10.4%. The results for the hypochlorite yield could be low from the fibers bonding back together after blending. However, if constant agitation could have been possible from the time of blending to the six cut screen, possibly the yield percentage could have been higher. See Graph 6.

Control II versus sodium hydrosulfite showed similar results to the hypochlorite. See Table 7.

Table 7

<table>
<thead>
<tr>
<th>Percent Yield</th>
<th>Control II (15 min)</th>
<th>Sodium Hydrosulfite 2.6%</th>
<th>Sodium Hydrosulfite 5.2%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>90%</td>
<td>61%</td>
<td>58%</td>
</tr>
</tbody>
</table>
The highest yield value from the sodium hydrosulfite of 61% resulted from the concentration of 2.6%, whereas the lower yield value of 58% resulted from the concentration of 5.2%. These values are low compared to the 90% yield from control II. When comparing the highest and optimum values for hypochlorite and sodium hydrosulfite, the hypochlorite resulted with a higher yield value of 63% compared to the sodium hydrosulfite of 61%. However, the values are not very different from each other. See Graph 7 and 8.
GRAPH 6: PERCENTAGE YIELD
CONTROL II VS HYPOCHLORITE

Control II (15 min)  Hvoochlorite 5.2%  Hvoochlorite 7.8%  Hvoochlorite 10.4%
GRAPH 7: PERCENTAGE YIELD
CONTROL II VS SODIUM HYDROSOULFITE

- Control II (15 min)
- Sodium Hydrosoulfite 2.6%
- Sodium Hydrosoulfite 5.2%
GRAPH 8: PERCENTAGE YIELD
SODIUM HYDROSULFITE VS HYPOCHLORITE

YIELD
%
63%
62%
61%
60%

Sodium Hydrosulfite 2.6%
Hypochlorite 5.2%
Tensile versus Yield

When comparing the values of hypochlorite for tensile strength and yield percentage, a linear scale is followed. See Table 8.

<table>
<thead>
<tr>
<th></th>
<th>Percent Yield</th>
<th>Tensile Strength</th>
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<tbody>
<tr>
<td>Hypochlorite 5.2%</td>
<td>63%</td>
<td>1.0677 kN/m</td>
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<tr>
<td>Sodium Hydrosulfite 5.2%</td>
<td>58%</td>
<td>0.7758 kN/m</td>
</tr>
</tbody>
</table>

As the value of tensile strength increases, the yield percentage decreases with the hypochlorite. The sodium hydrosulfite showed an increase with yield as tensile strength increased. However, the yield percentages for the sodium hydrosulfite show a difference of 3 %, thus not showing a dramatic change. Both hypochlorite and sodium hydrosulfite were used for comparison. However, at this point in the experiment the hypochlorite is tending to be the optimal chemical of choice over the sodium hydrosulfite for tensile strength and yield percentage results. See Graph 9 and 10.
GRAPH 9: TENSILE STRENGTH VS YIELD %
HYPOCHLORITE

YIELD % 67% 68% 69% 70% 71% 72%

TENSILE STRENGTH kN/m 0.75 0.80 0.85 0.90 0.95 1.00 1.05 1.10
GRAPH 10: TENSILE STRENGTH VS YIELD %
SODIUM HYDROSULFITE

YIELD %

TENSILE STRENGTH kN/m
Cost

The cost of the two reagents was compared as well. See Table 9.

<table>
<thead>
<tr>
<th></th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hypochlorite</td>
<td>$ 2.00/gal</td>
</tr>
<tr>
<td>Sodium Hydrosulfite</td>
<td>$11.50/kg</td>
</tr>
</tbody>
</table>

The cost of the hypochlorite was roughly around $2.00 per gallon. Whereas, the cost of the sodium hydrosulfite was $11.50 per kg. With comparing the percent yield, tensile strength, and cost, it is found that the use of hypochlorite is much more economical. See Graph 11.
GRAPH 11: COST
SODIUM HYDROSULFITE VS HYPOCHLORITE

Sodium Hydrosulfite

Hypochlorite

$12.00

$10.00

$8.00

$6.00

$4.00

$2.00

$0.00
VI. CONCLUSIONS

- Combination of mechanical shear and hypochlorite at 5.2% achieved the highest tensile strength.

- Control II resulted with 90% yield. This was the highest compared to all other runs.

- Hypochlorite proved to be the most effective reagent in testing tensile strength, percent yield, and cost.

- Soaking times and temperature proved to be effective variables of the experiment.

- Tensile strength decreased by 27% and 6% with overuse of hypochlorite and sodium hydrosulfite respectively.
RECOMMENDATIONS

There is an existing need to seek new technologies and chemistries for finding efficient ways to reducing energy consumption and the application of chemicals to secondary wet strength pulp. Further work could be done to determine the results of the use of various other reagents such as polyacrylamide and epoxins on breaking down bottle carriers. The use of intense steam, pressure and soaking time could be further explored as well. However, it would be interesting to investigate new innovations on adding wet strength to the paper products. Other wet strength additives may break down easier under steam and pressure.
REFERENCES


APPENDICES
APPENDIX A
TAPPI STANDARDS

Laboratory Processing of Pulp  T 200 om-85

Forming Handsheets for Physical Tests of Pulp  T 205 om-88

Freeness of Pulp  T 227 om-92

Physical Testing of Pulp Handsheets  T 220 om-88

Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products  T 402 om-88

Tensile Strength of Paper  T 456 om-87

Grammage of Paper and Paperboard  T 410 om-88