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The Use of Demolition Wood in Papermaking

Todd W. Anderson
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The Use of Demolition Wood In Papermaking

by

Todd W. Anderson

A Thesis submitted
in partial fulfillment of
the course requirements for
The Bachelor of Science Degree

Western Michigan University
Kalamazoo, MI
12 April 1996
ABSTRACT

The reason for this study came from three factors affecting the paper industry as well as society as a whole. These three factors are decreasing landfill space, increasing tipping fees, and increasing demands for alternative fiber sources. In the United States, approximately 9.8 million tons of demolition wood waste is landfilled per year. Increasing governmental regulations which require greater amounts of secondary fiber to be used in papermaking are forcing the industry to consider alternative fiber options. The seven steps in the procedure include: rough wood, wood chips, kraft pulping, fiber refining, screening, handsheets, and testing. The four types of wood involved are a 50 year old house wood, a 100 year old barn wood, a kiln-dried wood, and a red pine green wood. All wood types are pine. The strength tests (tensile, burst, tear) revealed that demolition wood fiber is strong enough to be considered as a secondary fiber source. The average value obtained for tear from the demolition wood is 13.7 (mN m²/g). The average values for burst and tensile are 20.3 psi and 4.18 km, respectively. In fact, the values obtained from the old wood are comparable to those obtained by Kleppe for a green wood pine(4). Drawbacks to its use include contaminant removal, probable bleaching limitations, obtaining the wood, and labor/energy requirements. The resource may best be utilized by having a demolition company deliver the wood, pulp, screen, and refine the wood separately, and have the fibers metered in at known quantities at the blend chest within the paper mill. Ultimately the use of this source becomes a function of society's focus on the green movement and regulations imposed on the industry by the government.

4/12/96
A SPECIAL THANKS TO THE FOLLOWING PEOPLE
FOR THEIR HELP AND GUIDANCE

Dr. David K. Peterson

my family and friends
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INTRODUCTION

The reason for this study came from three factors affecting the paper industry as well as society as a whole. The three factors are decreasing landfill space, increasing tipping fees, and increasing demands for alternative fiber sources.

Decreases in landfill space and increases in tipping fees have a direct relationship. This relationship follows the fact that as the available space for demolition waste storage decreases, the cost for dumping the waste in a storage facility increases. Typically, the majority of demolition waste is dumped at non-hazardous landfill sites. Annually, 315 pounds per capita of demolition waste is landfilled, twenty to thirty percent of which is wood waste (1). In the United States, that equates to approximately 9.8 million tons of demolition wood waste per year.

The third factor, increasing demands for alternative fiber sources, is a result of the nation's ongoing green movement as well as an industry response to increasing governmental regulations which require greater amounts of secondary fiber to be used in papermaking. The overall goal of this thesis is to determine if the demolition wood is a usable fiber for papermaking and to turn a societal liability into an asset. To achieve this goal, four different wood types including two demolition wood sources, a kiln-dried source, and a green source were chosen and used to draw comparisons. Each wood
type was a form of pine. For detailed explanations of wood sources and experimental procedures, please refer to experimental procedures section of this report.
THEORETICAL AND BACKGROUND DISCUSSION

It is a fact that never dried pulps or green wood pulps tend to exhibit better overall strength properties than fibers which have been dried and then re-hydrated. This is what is believed will happen with the demolition wood sources. The lengthy aging and drying imposed on these wood sources should make them very resistant to liquor penetration which will decrease their ability to form a usable sheet of paper.

Prior work done on demolition wood has not been directed at the paper industry. After using five different literature search mechanisms, it was determined that there was no literature available in this area of study. The search mechanisms used are as follows. The first search was done through Western Michigan University's library. Its resources were very limited in any area related to demolition wood. The second search was done through the Dow Chemical Co.'s science database. It revealed some articles on palletizing and its related uses but nothing directly related to papermaking. The third search was attempted through TAPPI's technical library. Unfortunately, this search revealed nothing. The fourth search was tried through the U.S Forest Products Society and revealed an article which discussed compressed chip boards made of demolition wood chips. This article was deemed unusable since it was not directly related to this study. The last attempt was directed at Longview Fibre in Washington. It is believed that the trees affected
by the Mt. Saint Helens eruption may exhibit characteristics similar to those of demolition wood. Unfortunately, no information was available.
EXPERIMENTAL PROCEDURES

Since the material available on this subject was so limited, the experimental design was left very open. The design was kept simple to minimize variations in the process among the various wood types. The seven steps in the procedure include: rough wood, wood chips, kraft pulping, fiber refining, screening, handsheets, and testing. In depth descriptions of the steps in the experimental design are as follows.

The first step was obtaining the various wood types. The green wood source came from Dr. Peterson's yard and was red pine. The kiln-dried wood source also came from Dr. Peterson but was of the variety of pine lumber which could be found at any lumber yard. The two demolition wood sources were much harder to obtain. Each consisted of a unknown type of pine. One source came from a fifty year old house located in Midland, MI while the other source came from a one hundred year old barn located in Kalamazoo, MI.

The second step in the procedure involved chipping the different wood sources. A backyard chipper, a Kemp TGCH, was used to chip the green wood. This method was also attempted on the other three sources. Due to the dryness of the wood, the shredder essentially pulverized the wood into saw dust. Since this was unacceptable, the remaining three sources were cut into uniform chips approximately one inch square with a
thickness of .1875 inches using a table saw. It should be noted that before the three sources could be cut, contaminants found in the demolition wood had to be removed. This procedure was very labor intensive.

Eight liters of pulping liquor were made and consisted of nineteen percent Na₂O. Na₂O measures the percentage of NaOH and Na₂S within the liquor. The liquor was made to specified standards obtained from the Paper 203 kraft liquor makeup procedure. Kraft liquor was chosen because it provides good strength to the pulp as well as having an adequate yield.

All pulping was performed in the M/K lab scale batch digester located in the pulping lab adjacent to the pilot plant. All chips except green were given a twenty minute presoak in the digester to aid in liquor penetration. Temperatures for each cook were raised to just over 170 degrees Celsius. The pressure within the digester was maintained at 100 psi. The digester was blown after a ten minute cool-down period which resulted in a total pressurized cooking time of fifty minutes. Once the chips were removed from the digester, the chips were washed and broken up in the British Disintegrator. Each chip sample was placed in the disintegrator for fifteen to twenty minutes. The total time per cook from uncooked chips to pulped fiber approximated three and a half to four hours. Three control factors were used in each pulping run. The first was H-factor which represents the relative reaction
rate versus time. Monitoring the H-factor allows for continuous evaluation of the reaction rate of the liquor with the pulp as well as allowing each pulp to be cooked to a similar, final H-factor. A detailed explanation of H-factor may be located in the Paper 203 pulping procedure. The second control factor was the temperature. The maximum temperature was maintained slightly above 170 degrees Celsius. At temperatures approaching 180 degrees Celsius, degradation and burning of the cook may occur. At temperatures lower that 170 degrees, the penetration may not be adequate enough to achieve proper lignin removal (2). The final factor was determining the alkali consumption within the cook. Knowing the charge of the liquor entering the cook and then utilizing a black liquor analysis after the completion of the cook allows for consumption comparisons of each cook. The black liquor analysis was performed according to standards found in the Paper 203 black liquor analysis procedure. The only variation entailed using a buchner funnel and filter pad to achieve a solids-free supernatant instead of using a centrifuge. The results of the Black Liquor analysis may be found in the results section of this report.

Refining was performed in the valley beater with ten pounds of weight on the load arm. Each pulp was beaten to an approximate freeness of 700 mL. One mL of Tamol 850 defoamer
was added to each pulp to help reduce large amounts of foaming which occurred during the refining stage.

The next step entailed screening of the refined pulps. Each pulp was screened using the six-cut screen located by the recycle area in the Pilot Plant. Accepts from the six-cut were collected in a one-hundred mesh screen. An accepts yield was then determined.

The accept pulp was then used to make three gram handsheets on the Noble and Wood handsheet maker. Five sheets of acceptable weight were made per pulp. One wet press was used with ten pounds of loaded weight.

The handsheets were conditioned in the testing lab overnight. Weight for each sheet was again determined as well as caliper. This was used to determine the density of the sheet. Strength tests were then performed on the sheets. These tests included: tensile, tear, and burst. The tensile test may be defined as the force required to break a narrow strip of paper where both the length of the strip and loading are specified. This test is a function of both fiber and bond strength and is a good measure of durability. The tear test involves a falling swing arm which continues a pre-cut tear in the sheet when a force is applied perpendicular to the sheet. This test is a good measure of fiber strength. A five sheet pad was used when this test was performed. The
burst test is a measure of the hydrostatic pressure necessary to rupture a flat sheet clamped in the apparatus. This test indicates toughness of the sheet and is often used in quality control. The three strength tests were performed according to TAPPI standard T 220 om-88. The final test was performed using the Kajaani FS-100 fiber length analyzer. Dilute fiber samples were placed into the analyzer, as they passed through the photoelectric optic cell, the portion of the light blocked by the fiber was measured as the fiber's length and recorded. Three to five thousand fibers were measured per run.
RESULTS PRESENTATION

Graphical representation and calculations for all the data discussed in this section may be located in the appendix section of this report.

The final H-factor, one of the important controlling variables in the pulping procedure, was at a relatively even level for each of the pulping runs. Kiln dried wood had a H-factor equal to 326 while the house, barn, and green woods had H-factors of 341, 381, and 371 respectively.

The black liquor analysis was performed and liquor consumption was determined. By determining the corresponding value in mL of .5 N HCl used to reach a pH of 8.3, the percentages of residual active alkali at the end of the cook and the percentage alkali consumed during the cook can be determined with a simple calculation. It was determined that the percentage active alkali at the end of the cook for the kiln dried wood, house wood, barn wood, and green wood was 20.5, 21.1, 22.8, and 8 percent respectively. The percentage active alkali consumed for the same woods was 59.2, 57.9, 54.5, and 84.1 percent respectively.

Respective freenesses for the kiln dried, house, barn, and green woods measured 705, 705, 710, and 700 mL respectively.
Accept yield from the six-cut screen resulted in 29.7 percent for the kiln dried wood, 22.1 percent for the house wood, 10.65 percent for the barn wood, and 8.7 percent for the green wood.

Physical testing of the three gram handsheets resulted in the following data. For each figure corresponding to the physical test, standard deviations for each value are located next to wood types in parentheses. The values for handsheet density are as follows: kiln dried, .284 g/cm³; house, .272 g/cm³; barn, .275 g/cm³, and green, .226 g/cm³. The values for tensile, in the previous order, are: 4.54 km, 2.85 km, 5.15 km, and 1.53 km. The values for the five sheet tear pad are: 15.3 (mN m²/g), 12.3 (mN m²/g), 13.5 (mN m²/g), and 9.8 (mN m²/g). The values for bursting strength in psi are as follows: 22.2, 13.2, 25.6, and 5.8. The average values for fiber length obtained from the Kajaani fiber length analyzer were measured in millimeters. The values for the kiln dried, house, barn, and green woods are as follows: 1.93, 1.90, 1.82, and 1.43 respectively.
DISCUSSION OF RESULTS

The H-factor values determined for each pulping run are close enough so that their small differences would have a minimal impact in causing pulp variability. Freeness values obtained from the beater runs are also close enough so that they would not play a significant role in sheet property variance.

Active alkali consumption values obtained from the black liquor analysis yielded results that were expected. While the green wood consumed approximately 84 percent of the active alkali, the other woods consumed, on average, only 57 percent. This follows theory in that the old, dry wood fibers are very resistant to pulping liquor penetration. The lack of penetration was also seen when the chips were being broken into fibers in the disintegrator. The green chips broke easily into fiber while many of the chips in the other three species were very resistant. After twenty minutes in the disintegrator, there were still many 'knots' present. This is a drawback to the demolition pulps because it reduces pulping yield.

Accepts yield on the six-cut screen was relatively opposite of what was thought would happen. The green produced the lowest accepts ratio at 8.7 percent. The other accept ratios were relatively low but were higher than those found in the green wood. It was apparent by physical examination that the green fibers were clumping together and plugging the screen.
This may be the reason for the low accepts result. The demolition wood sources rejects were mainly fiber bundles in which the pulping liquor had made successful penetration on the ends but did not reach the middle of the fiber bundle.

The results obtained from the physical testing were opposite of what was expected. Burst, tensile, and tear yielded results indicating that the green wood produced the weakest sheet of paper. It was believed that the old wood fibers would be very resistant to bonding and would produce a weaker sheet. Several reasons may be attributed to the low strength values found in the green wood. The first reason is bulking. Bulk is the reciprocal of density. The green wood was found to have the lowest density, therefore, it had the greatest bulk out of the four different sheets. The high bulk may be a function of fiber re-hydration due to conditioning and increased void spaces. If both of these situations occurred, fiber strength would decrease due to a reduced fiber to fiber bonding area. The second reason is fiber length. Through fiber length analysis, it was revealed that the green wood had the shortest average fiber length. Since longer fibers characteristically produce stronger sheets, the shorter fibers found in the green wood may have hurt its strength values when compared to the other pulps. The final reason is lignin content. A kappa number analysis was to be performed on each pulp to determine the actual lignin content of each species. Lignin found between the wood fibers drastically
decreases their bondability and results in strength loss. An assumption may be made that the green wood initially contained significantly higher amounts of lignin. The higher lignin levels would necessitate an increased pulping time at the maximum temperature level to remove the excess lignin. Since this was an unknown factor, adjustments in the cook were not made and higher lignin levels may be present in the green wood handsheets. Although the green wood results were unexpected, the positive strength properties found in the other three pulps should not be discounted. In fact, the values obtained from the old wood are comparable to those obtained by Kleppe. Kleppe ran a kraft pulping experiment and tested the strength properties of a green wood southern pine and obtained strength values slightly better than those obtained for the demolition wood species in this experiment. It must be noted, however, that Kleppe's pulps were refined to a freeness of 600 instead of 700 as in this experiment (4).
CONCLUSIONS AND RECOMMENDATIONS

The use of demolition wood as an alternative secondary fiber source in papermaking is very plausible. Demolition wood fibers have been proven to provide adequate strength in a sheet of paper. Drawbacks to its use include contaminant removal, probable bleaching limitations, obtaining the wood, and labor/energy requirements. The resource may best be utilized by having a demolition company deliver the wood, pulp, screen, and refine the wood separately, and have the fibers metered in at know quantities at the blend chest within the paper mill. Ultimately the use of this source becomes a function of society's focus on the green movement and regulations imposed on the industry by the government.
LITERATURE CITED


H FACTOR

RELATIVE RATES

390.00
380.00
370.00
360.00
350.00
340.00
330.00
320.00
310.00
300.00
290.00

KILN DRIED
HOUSE
BARN
GREEN

WOOD TYPES

324
274
381
370
PERCENT ACTIVE ALKALI CONSUMED

KILN DRIED: 59.76%
HOUSE: 52.9%
BARN: 54.5%
GREEN: 84.1%
PERCENT OF ACTIVE ALKALI AT END OF COOK

WOOD TYPES

KILN DRIED
HOUSE
BARN
GREEN

PERCENT

0.00
5.00
10.00
15.00
20.00
25.00

20.5
26.1
22.0
8
CANADIAN STANDARD FREENESS

WOOD TYPES

KILN DRIED

HOUSE

BARN

GREEN

705

705

710

700

702

704

706

708

710

694

696

698

700

702

704

706

708

710

mL
HANDSHEET DENSITY

WOOD TYPES

KILN DRIED (.005)  HOUSE (.008)  BARN (.023)  GREEN (.009)

DENSITY (g/cm³)
TEAR STRENGTH

KILN DRIED (.43)  HOUSE (.83)  BARN (1.12)  GREEN (.83)

WOOD TYPES

TEAR (mN m^2/g)

16  14  12  10  8  6  4  2  0
BURST STRENGTH

WOOD TYPES

KILN DRIED (2.61)  HOUSE (1.16)  BARN (3.47)  GREEN (0.65)

BURST (PSI)
TENSILE STRENGTH

WOOD TYPES

KILN DRIED (.704)  HOUSE (.308)  BARN (1.07)  GREEN (.351)
AVERAGE FIBER LENGTH

LENGTH (mm)

KILN DRIED  1.23
HOUSE       1.9
BARN        1.82
GREEN       1.43

WOOD TYPES
BLACK LIQUOR ANALYSIS - KILN DRIED WOOD

The graph shows the relationship between pH and the volume of 0.5 N HCl. The pH decreases as the volume of the acid increases, indicating a reaction likely involving the neutralization of a base in the black liquor. The graph suggests the point of equivalence, where the pH starts to drop rapidly with an increase in acid volume.

The handwritten notes on the graph read: "used in calc. A. Heali Conroy."
BLACK LIQUOR ANALYSIS - HOUSE WOOD
BLACK LIQUOR ANALYSIS - BARN WOOD

The graph shows the pH levels plotted against the volume of 0.5 N HCl added. The pH decreases significantly as the volume increases, indicating a strong acid-base reaction.
BLACK LIQUOR ANALYSIS - GREEN WOOD

The graph shows the change in pH over the addition of 0.5 N HCl. The pH decreases significantly as the volume of HCl increases, especially after approximately 10 ml. The curve indicates a sharp drop in pH, suggesting a strong reaction with the sample.
APPENDIX II
(PROCEDURES)
LAB DAY ONE

Calculations

A. Chip Charge

\[
\text{moist wood} = \frac{700 \text{ g O.J. wood} \times \frac{100}{100} \times \frac{109.4 \text{ g A.O. chips to charge}}{109.4 \text{ g A.O. chips}} - \frac{100 \text{ g O.J. chips}}{700 \text{ g O.J. wood}}} = 394 \text{ g A.O. chips}
\]

B. Liquor Charge

\[
\text{1338 \text{ ml liquor to charge}}
\]

C. Water Charge

\[
\text{ml total liquid} - \text{ml water in chips} - \text{ml liquor added} = \text{ml water to add}
\]

\[
\frac{700 \text{ g O.J. wood} \times \left( \frac{19}{100} \right) \left( \text{Na}_2\text{O on chips/100} \right) \times (1000 \text{ ml/l})}{\left( \frac{19}{100} \right) \left( \text{Na}_2\text{O in liquor} \right)} = 1329 \text{ ml liquor to charge}
\]

OPERATING PROCEDURES

Weigh \( \frac{700 \text{ g O.J. chips}}{1000 \text{ g O.J. wood}} \) of O.J. chips in the metal basket. You will probably have to pack the chips by hand. Be careful not to rip the screen in the bottom of the basket.

Close the bottom drain valve below the pump.

Add the calculated amount of water. Carefully add the calculated amount of cooking liquor.

Close the circulating valve at the top of the digestor almost shut. Never run the pump with the circulating valve completely closed as this will damage the pump.

Start the pump. Check to be certain that the water is circulating. It may be necessary to drain some water from the bottom to prime the pump. Starting and stopping the pump several times may also help.

Place the perforated cover plate on the chips. Place the basket of chips in the digestor. Be certain that the cut-out section of the basket is beside the circulating line opening.

Place the cover on the digestor. Have the valve open and the vent pipe at the side of the apparatus. Tighten the thumb nuts finger tight. Do not overtighten! Tightening the nut down with a wrench may cause the bolts to fail under the high pressure of the cook.
OPERATING PROCEDURES (Cont.)

Completely open the circulating valve.

Turn on both the controlled and continuous heaters. Record the time. Set the controlled heater at the desired temperature. Set the power rheostat at the 170° mark.

Place a metal beaker under the vent line. Watch for liquid discharge from the vent line into the beaker when the digester approaches 100°C. Close the vent line valve to stop this discharge and to begin pressure buildup.

When the cook reaches the desired temperature, turn off the continuous heater and start timing the cook. Record the time required to reach temperature. Turn the continuous heater back on (or increase the power rheostat) if the controlled heater is unable to hold the proper temperature.

Upon completion of the cook, turn off the heaters. Turn off the pump. Close the circulating valve. Do not attempt to restart the pump during the pressure relief/cooling cycle. Doing so will cause it to cavitate and run air-bound.

Place a clean metal beaker under the vent line. Cover it with aluminum foil.

Carefully open the vent line valve part way and allow the pressure to slowly push the liquor into the metal beaker until the pressure is relieved. The digester should be close to atmospheric pressure upon completion of this operation.

Remove the top lid. Using gloves, remove the basket of cooked chips. Save a quart of black liquor from the metal beaker for testing next lab day.

Rinse the digester and pump thoroughly with water.

PROCESSING OF M/K COOKED PULP

Place the basket of chips in the sink and run cold water over the chips to cool them and remove surface black liquor. Continue running cold water for 2-5 minutes.

Using rubber gloves, transfer 1/5 to 1/4 of the cooked chips to a large Waring Blender. Add water and break up the chips by running for 3-5 minutes at low speed.

Repeat with the other portions of chips. With softwood, a small amount of defoamer may be necessary.

Position the stand with the cloth bag under the outlet of the vibrating laboratory flat screen. This screen should have a 0.014" slotted plate.

Combine the screening and washing procedures by screening small portions of pulp with continual water addition.
PROCESS OF M/K COOKED PULP (Cont.)

Collect the screen rejects and place them in a tared weighing dish. Dry them in the oven. Report the "percent screen rejects" based on the original O.D. weight of chips.

Collect the screened pulp in a cloth bag. Partially dewater it by hand. Using the pulp-centrifugal extractor, dewater the pulp to approximately 27% O.D. fiber.

Remove the pulp from the cloth bag and place it in a tared plastic bag. Obtain the weight of the pulp.

With the pulp in the plastic bag, mix it thoroughly by hand. Select two representative 20g samples. Place the samples in tared aluminum pans and dry overnight. Save the rest of the pulp in the plastic bag. Make sure that the bag is clearly labeled.

Place the clean baskets back in the clean digesters. Be certain that the perforated plates are in the baskets. Place the cover on the digester and loosely put on all thumb nuts.
Appendix B1 Discussion of H-Factor

8.38 Temperature of Digestion

Temperature and time are interdependent variables; the higher the temperature the less the time of digestion. A temperature below 170°C apparently gives no advantage in either yield or quality. Above 180°C the cellulose becomes increasingly susceptible to degradation, and particularly so at a temperature of 190°C. Temperatures of 170 to 180°C (338 to 356°F) are thus common in commercial practice. In batch cooking, the liquor is introduced into the digester at a temperature, typically 10 to 80°C (50 to 140°F), below the boiling point of water, and the contents of the digester are then heated to the predetermined, constant, cooking temperature. The rise to temperature is carried out as rapidly as possible within the limitations of the equipment and the available steam pressure, but, owing to the size of the operation, this means, in practice, that the time of rise is fairly gradual—1.5 to 2 hr being common. A cooking cycle that consists of a rise to temperature period and a period at temperature.

8.31 The H-Factor

In 1931 Vonnegut developed a method of expressing cooking times and temperatures as a single variable so that the times and tempera-

8.29 RELATIVE RATES

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In 1931 Vonnegut developed a method of expressing cooking times and temperatures as a single variable so that the times and tempera-
Table A.2.1 is not legible but appears to be a table of data. The text refers to Table A.2.1 and mentions that it contains steam pressures and corresponding temperatures, but the specific values are not readable.

The curve of temperature vs. time, with its corresponding curve of relative rate vs. time, is shown in Figure 8.67. The II factor, i.e., the area under the rate curve, is given by the total value in column 6 of Table A.2.4, obtained by adding the products of average relative rates and the corresponding time intervals over which the average rate persists. When the data obtained by Haggland and Hedlund for pitch-cooked spruce at three different temperatures in liquid at 31% sulphidity are plotted against the II factor for each cook, a single relationship (Fig. 8.60) is obtained for both yield and lignin content. The series at 10% sulphidity showed similar concordance, but for zero sulphidity, i.e., soda liquor, a value of 35,000 cal/mole must be employed for the activation energy, leading to a value for A of about 17.810, in order to fit the lower relative rates of delignification.

Application. The II factor is not only of value in correlating research results but is also extremely useful in mill operation. It enables changes to be made in the time temperature cycle while maintaining a constant degree of delignification. Also, if precise control of the cycle is not possible for any reason, the II factor can be calculated as the cook proceeds and the desired total II factor can be achieved by adjusting the time of blow. Integrators which provide a continuous reading of II factor are commercially available.

As will be seen from Table A.2.1 and from the graph of relative rate vs. temperature (Fig. 8.60) the rate of pulping increases by a factor of about 3.3 for each 10°C increase above 170°C. Although it is true, as a consequence of this factor, that the reaction rate is
### Table 8.11: Calculation of the II Factor, Digestion at 170° C Calculation of Total Time Required to Achieve Identical II Factor When Temperature Ranged to 100°

<table>
<thead>
<tr>
<th>Time from start, hr</th>
<th>Temp., °C</th>
<th>Rel. reaction rate</th>
<th>Avg. rate</th>
<th>X</th>
<th>Time, hr</th>
<th>II°</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>90</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>0.13</td>
<td>0</td>
</tr>
<tr>
<td>0.15</td>
<td>95</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>0.15</td>
<td>1</td>
</tr>
<tr>
<td>0.50</td>
<td>110</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>0.15</td>
<td>1</td>
</tr>
<tr>
<td>0.75</td>
<td>120</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>0.15</td>
<td>1</td>
</tr>
<tr>
<td>1.00</td>
<td>150</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>0.08</td>
<td>10</td>
</tr>
<tr>
<td>1.15</td>
<td>155</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>0.08</td>
<td>11</td>
</tr>
<tr>
<td>1.50</td>
<td>170</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>0.08</td>
<td>11</td>
</tr>
<tr>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td></td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>2.00</td>
<td>170</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>1.50</td>
<td>1,500</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Total, whole cycle = 1,500</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.00</td>
<td>90</td>
<td>0</td>
<td>X</td>
<td>0.08</td>
<td>10</td>
</tr>
<tr>
<td>1.15</td>
<td>110</td>
<td>0</td>
<td>0</td>
<td>X</td>
<td>0.15</td>
<td>11</td>
</tr>
<tr>
<td>1.67</td>
<td>100</td>
<td>0.007</td>
<td>0.104</td>
<td>X</td>
<td>0.15</td>
<td>11</td>
</tr>
<tr>
<td>2.00</td>
<td>180</td>
<td>0.057</td>
<td>0.057</td>
<td>X</td>
<td>0.15</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Total, whole cycle = 1,500</td>
<td></td>
</tr>
</tbody>
</table>

*Calculated for the nearest whole number. Relative reaction rates are taken from Table 8.1, page 442.

more than doubled by increasing the temperature by 10° C, this does not mean that the total digestion time can be halved. At the higher temperature a proportion of the total time is required to bring the digester to the increased temperature and during this period, cooking proceeds at an accelerating rate. It should be noted that Haggland and Hedlund (Figs. 8.51 and 8.53) took 8 hr to bring the digester to temperature, regardless of the final temperature, and that, in certain of the examples cited, this time is approximately 80% of the total time.

In commercial practice the digester is heated as rapidly as possible within the limitations of the equipment and the available steam pressure, so that it takes the same time to reach 100° C regardless of whether this is the final temperature or it is raised a further 10° C. Other wood will be pulped to the same extent if a temperature of 100° C is maintained for a similar time. In considering two digestions, at 170° and 100° C maximum temperature, it is the time at 170° C or higher that should be taken into consideration. Assuming that it takes 1.5 hr to bring the cook to 170° C, and 1.5 hr at that temperature for a given degree of pulping—a total of 3 hr, as with the cooking cycle illustrated in Figure 8.67—the II factor (Table 8.24) is 1,552. Assuming that the rise from 170 to 180° C takes 10 min, a total contribution of 450 will have been made to the II factor by the time the cook reaches 180° C. Subtracting this from 1,552 leaves 1,102. At the relative rate of 2,057, which persists at 180° C, an additional time of 1,102/2,057 = 0.54 hr at 180° C will be needed to reach the same total II factor (Table 8.24) and hence the same degree of delignification. The total time is then 3.18 hr. Thus the actual saving in cooking time is only 30%. Greater savings in time can be achieved if the cook
BLACK LIQUOR ANALYSIS (cont.)

Preparation of Liquor

Pipet 2 50.0 ml samples of black liquor into a 500 ml volumetric flask containing approximately 100 ml of distilled water.

Add 200 ml of 0.5 M BaCl₂.

Fill to the mark with distilled water and mix well.

Pour into four 50 ml centrifuge tubes. Make certain that the tubes are dry or have been rinsed with a small amount of the liquid to be centrifuged. Be certain that opposite tubes have the same amount of liquid for proper balance.

Centrifuge until the supernatant liquor is clear (at least five minutes).

Pour the clear supernatant liquid into a clean dry 250 ml beaker.

Procedure

Pipet 100.0 ml of the supernatant liquid into a dry 250 ml beaker.

Insert a magnetic spin bar. Add 5 ml of 40% formaldehyde.

Titrate with 0.500 N HCl to a pH of 3.5. Follow the course of the reaction potentiometrically with a pH meter. Add the 0.500 N HCl in increments of fixed volume and record the pH value after each addition.

For pH 10 and above, add 2.00 ml increments of .500 N HCl.
For pH 9-10, add 1.00 ml increments of .500 N HCl.
For pH 6-9, add .20 ml increments of .500 N HCl.
For pH 5-6, add 1.00 ml increments of .500 N HCl.
For pH below 5, add 2.00 ml increments of .500 N HCl.

Plot the data with pH as the y-axis and milliequivalent of HCl as the x-axis. Connect the points in a smooth curve.

Obtain the milliequivalent of acid corresponding to the end point by projecting the inflection point at pH 8.3 to the x-axis.

From this value subtract the acid equivalent, if any, of the 5 ml of formaldehyde. Determine the acid equivalent of the formaldehyde, using phenolphthalein as an indicator. Use a 50 ml Erlenmeyer flask and titrate with the same 0.500 N HCl. The instructor will give this information.

Calculations

\[
\text{meq of .5 N HCl} = \text{meq of .500 N HCl for sample} - \text{meq of .500 N HCl for formaldehyde}
\]

\[
g/1 \text{ Active Alkali as Na}_2O = \text{meq of acid} \times 1.55
\]