

## IMPACT OF IMPREGNATION ON HIGH KAPPA NUMBER HARDWOOD PULPS

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Mixed US southern hardwood chips were impregnated and cooked under various conditions in the laboratory. Kappa numbers between 15 and 55 were obtained. The reject content, kappa number, and yield were studied as a function of time, temperature, and alkali charge in the cooking. The results indicate that by using modern cooking technology, i.e. CompactCooking™, an optimal point with respect to the studied pulp parameters can be found. Selected pulps were bleached to high brightness with several different bleaching sequences. These pulps were beaten in a PFI mill and their physical properties were compared at a constant bulk. High kappa, well impregnated fully bleached pulps exhibited improved yield with equivalent or better physical properties as compared to conventional kraft pulps. The high kappa, well impregnated pulps were also found to have improved bleachability as compared to conventionally cooked, bleachable grade kappa number pulps.

*Keywords: Hardwood; High kappa; Impregnation; Compact cooking, GI cooking; Bleaching; Enzymes; Physical properties*

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### INTRODUCTION

With the implementation of oxygen delignification, it has been possible to increase the target kappa number of the pulp without substantially increasing the total bleach plant effluent loading or increasing the bleaching costs (McCubbin 1997). The main benefit of increasing the cooking target kappa number is an increase in the final beached pulp yield (Hart and Connell 2006). An increased bleached pulp yield allows the mill to improve the utilization of wood by converting more of this raw material into a higher value product, i.e. pulp, instead of converting it into energy via black liquor combustion. An increased pulp yield decreases production costs per ton of pulp and potentially increases the production volume, since the load on the recovery boiler is decreased. Frequently, the recovery boiler load is the production bottleneck in many mills (Kirkman et al. 1986).

Despite all the cost and sustainability benefits obtained from increasing the cooking kappa number target, there is a technical limit to how high of a kappa number may be successfully achieved. As the kappa number increases, a point is reached where the reject levels in the resulting pulp will increase dramatically (Hart 2011). This point, called the defibration or fiber liberation point, will usually limit the maximum kappa number target in a mill. Frequently, a mill will purposely operate with a digester kappa

number target setting at least 3 to 5 kappa units below this point to allow for process upsets without exceeding the defibration point. It has been shown in laboratory cooking that by extending the impregnation portion of the cooking profile, the defibration point of both softwood (Karlstrom 2009) and hardwood (Nasman et al. 2007; Wedin et al. 2010) kraft pulps can be significantly increased to higher kappa numbers, e.g. about 90 kappa for softwoods and 30 kappa for hardwoods.

It has been suggested that softwoods tend to defiberize at higher kappa numbers than hardwoods because of the higher concentration of guaiacyl lignin in the middle lamella of softwood fibers as compared to hardwoods (Lindstrom 2011). The guaiacyl lignin reacts more slowly than syringyl lignin (Christiernin 2006; Santos et al. 2011). Thus, the syringyl lignin in the hardwood middle lamella will react faster than the guaiacyl lignin, resulting in a lower lignin content in the hardwood pulp before the fibers are chemically separable as compared to softwood pulps.

CompactCooking<sup>TM</sup> is a modern cooking technology that considers the importance of the impregnation portion of the kraft cook (van Tran 2005) to produce a high quality pulp with a lower than standard reject content. In CompactCooking<sup>TM</sup> G2, the impregnation stage has been further improved through prolonging the impregnation stage at a lower temperature in order to promote diffusion over consumption of cooking chemicals during the impregnation stage of the cook (van Tran 2002).

It has been well documented that a major source of cooking yield loss results from nonuniform cooking of the pulp fibers (Scallan 1992). Nonuniformity has also been shown to be a cause of strength loss in the resulting pulp (Tichy 1981). A major source of nonuniformity in kraft cooks has been traced to mass transfer limitations (Jimenez et al. 1990). Temperature has a significant impact upon both the diffusion rate and reaction rate of kraft cooking chemicals (Sixta 2006). As the temperature in the early portion of the cook increases, significantly more alkali is consumed by chemical reaction at the expense of chemical diffusion into the fiber (Hultholm 2004). As the rate of alkali consuming reactions is highest at the beginning of the cook, increased temperatures in the impregnation stage will significantly reduce the amount of alkali available for diffusion (Courchene et al. 2005).

The mass transfer restrictions in the kraft pulping process can be limited by employing an impregnation stage, which allows the cooking liquor to penetrate into the chips before the temperature is raised to cooking conditions. Impregnation and cooking uniformity has been reported to reduce the amount of rejects produced in the unbleached pulp (Tolonen et al. 2010; Malkov et al. 2002). Good impregnation has also been reported to reduce the total H-factor required to achieve a target kappa number in the cooking process (Malkov et al. 2002).

Recently, a novel method of pulp preparation with enhanced yield and pulp quality has been developed (Hart et al. 2008a). The new pulping method breaks the current pulping paradigm of pulping to a low reject level. This process purposefully stops the kraft pulping process at abnormally high kappa numbers and high reject levels. By terminating the kraft cook at substantially higher than traditional kappa numbers, a defibrated portion of the cook can be separated in the screening process. This high kappa fiber has excellent physical properties. The undefibrated portion of the cook is then mechanically treated in a high consistency refiner. The resulting refined pulp is screened,

with the accepts portion being combined with the accepts from the first screening process or used as a separate fiber stream (Hart et al. 2008b). The resulting combined accepts streams may be used as a brown fiber or bleached to high brightness. Excellent bleached pulp physical properties equal to or better than conventional kraft cooking have been achieved in pilot scale investigations employing this process (Hart 2011).

The current work examines the impact of the extended time, low temperature impregnation process associated with the CompactCooking™ process upon the higher kappa number kraft pulps utilized with this novel cooking process. By minimizing the amount of undefibrated fiber in the high kappa cooking process, improved pulp yield and physical properties should be obtained. Ideally by employing the improved chip impregnation available with the CompactCooking™ process, a lower percentage of rejects would be realized at the higher kappa numbers and improved pulp yield and strength properties will be realized.

CompactCooking™ was simulated in the laboratory by applying different cooking parameters in order to minimize the reject levels obtained at the higher kappa numbers. The laboratory residence times for each stage of the cooking process were set corresponding to the existing two vessel continuous digester at the MWV Evadale, TX mill.

### **Retrofit of an EMCC™ Digester to CompactCooking™**

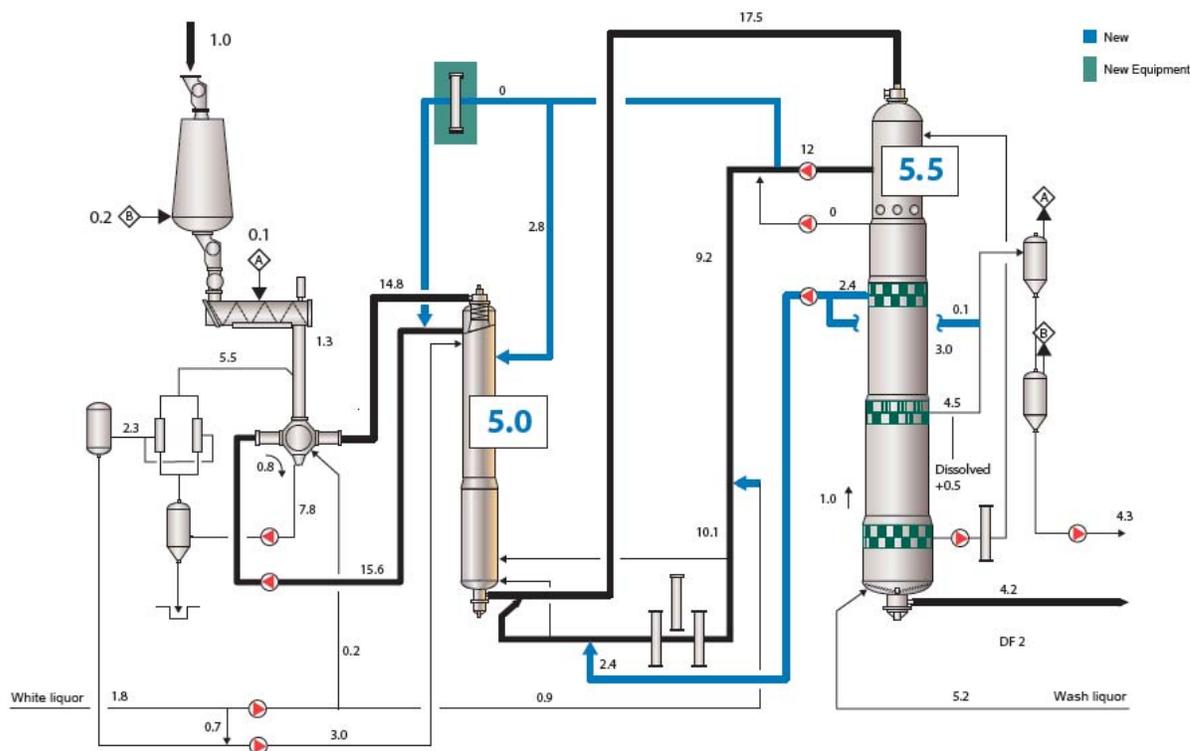
A significant part of the CompactCooking™ philosophy is to maintain a high liquor to wood ratio (L:W) and a high sulfidity in the impregnation portion of the cook. In order to obtain these conditions, black liquor is taken from the upper cook extraction zone of the digester and circulated back into the impregnation vessel and into the bottom circulation (BC) loop. Figure 1 shows a potential method of retrofitting the existing Evadale TX digester for the new cooking process. The blue lines in Fig. 1 represent new piping changes required to perform this conversion.

This new configuration would increase the L:W in the impregnation stage of the digester to approximately 5.0 with a residence time of about 18 minutes. The first cook zone of the digester would have a L:W of about 5.5 with a residence time of about 109 minutes. The use of black liquor obtained from the main extraction zone of the digester to the BC loop ensures a high sulfidity in the impregnation and top cooking portion of the cook.

## **EXPERIMENTAL**

### **Materials**

Mixed US Southern Hardwood chips from the Evadale, TX mill, USA were used in this study. The chips were screened through holes and only the accept fraction retained on the 3 mm holes and passing through 45 mm holes ( $>\text{Ø}3\text{mm} < \text{Ø}45\text{mm}$ ) were used in the trials. A mill cooked pulp was obtained from similar raw material for bleach comparisons.



**Fig. 1.** Potential method of retrofitting the existing two-vessel hydraulic digester at Evadale TX to perform the new cooking method. Note blue lines are new piping. The numbers represent L:W in the various zones of the digester. The residence time in the impregnation stage of the cook would be about 18 minutes for this digester configuration.

## Cooking

With the current cooking system at Evadale mill (a two-vessel hydraulic digester) in consideration, different cooking conditions were evaluated. The laboratory cooking conditions were developed to simulate the industrial process design as shown in Fig. 1. The cooks were performed in a forced circulation digester with a volume of 15 L and a capacity of about 2 kg of chips. Impregnation time, temperature, L:W and alkali charge were kept at 18 min, 275°F (125°C), 5.0 and 22g/L, respectively. Cooking time was varied between 110 and 302 minutes and temperature between 266 and 293°F (130-145°C).

## Bleaching

Laboratory bleaching of selected lab produced cooks was performed to determine the impact of oxygen delignification with and without the addition of 0.3 Kg/ODT xylanase enzymes (EC 3.2.1.8, BioBrite® UHB, Iogen Corp., Canada) prior to D(EP)DD bleaching. The D, (EP), and X stages were performed in sealed plastic bags in a water bath. The O stage was performed in teflon lined stainless steel autoclaves rotated in a heated polyethyleneglycol bath using 26 kg/ODT caustic and 0.5MPa oxygen pressure. All bleaching stages except the enzyme stage were thoroughly washed after completion.

The general conditions for each bleaching stage are shown in Table 1. Selected bleached pulps were beaten with a PFI mill and subjected to physical property testing.

### Analysis

The cooked pulp was defibrated in a propeller disintegrator. Unscreened pulp yield was determined after washing and thickening. Thereafter, pulps were screened in two stages. First the coarse material was removed in a NAF (Nordiska Armatur Fabriken, Sweden) water jet defibrator with a perforation of 2.0 mm, at water pressure head of 150 kPa. A subsequent vibrating flat screen with 0.15 mm slots separated residual shives. The experimental process and the resulting pulps, both brown and bleached, were analyzed according to the methods listed in Table 2.

**Table 1.** General Bleach Stage Conditions

Stage	O	X	Do	(EP)	D1	D2
Consistency %	12	10	10	12	12	12
Time, min	60	60	60	60	120	120
Temp, °C	~100	55	55	80	Varied	Varied
Pressure, MPa	0.5	---	---	---	---	---
Terminal pH	~ 10.5-11.0	~6.5-7.0	2.5-2.8	>10.5	3.2-3.5	3.8-4.2

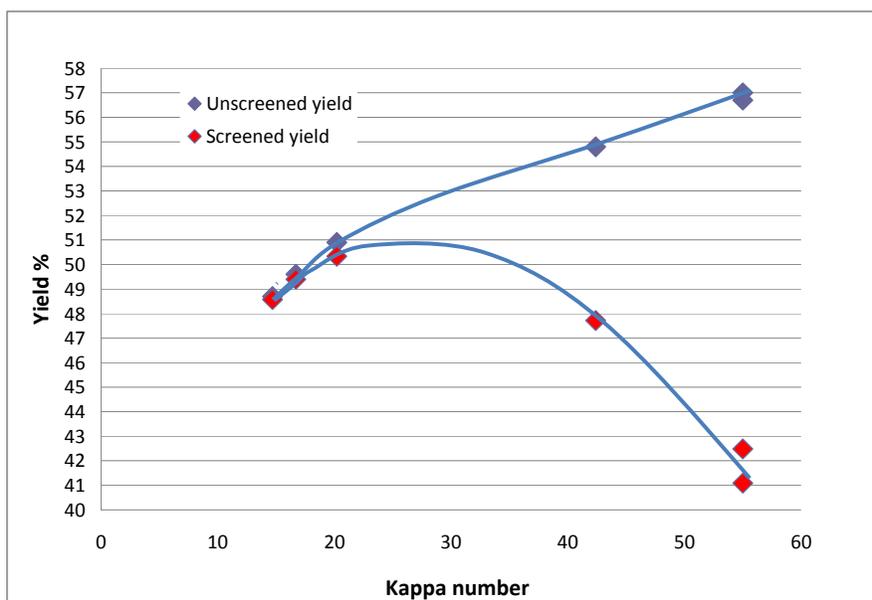
**Table 2.** Methods Used in the Study

Analysis	Method used
Kappa number	ISO 302:2004
Sheet forming	ISO 5269-1:1998
Thickness	ISO 534:2005
Grammage	ISO 536:1995
Tensile Properties	ISO 1924-3:1994
Tear index	ISO 1974:1990
Gurley	ISO 5636-5:2003
Intrinsic viscosity	ISO 5351:2004
Brightness, % ISO	ISO 2470:1999
Brightness reversion	4 hr @ 105 C (TAPPI UM 200)
Fiber dimensions	TAPPI T271 om-02
PFI mill beating	ISO 5264-2:2002
Resistance to bending	ISO 2493:1992

## RESULTS AND DISCUSSION

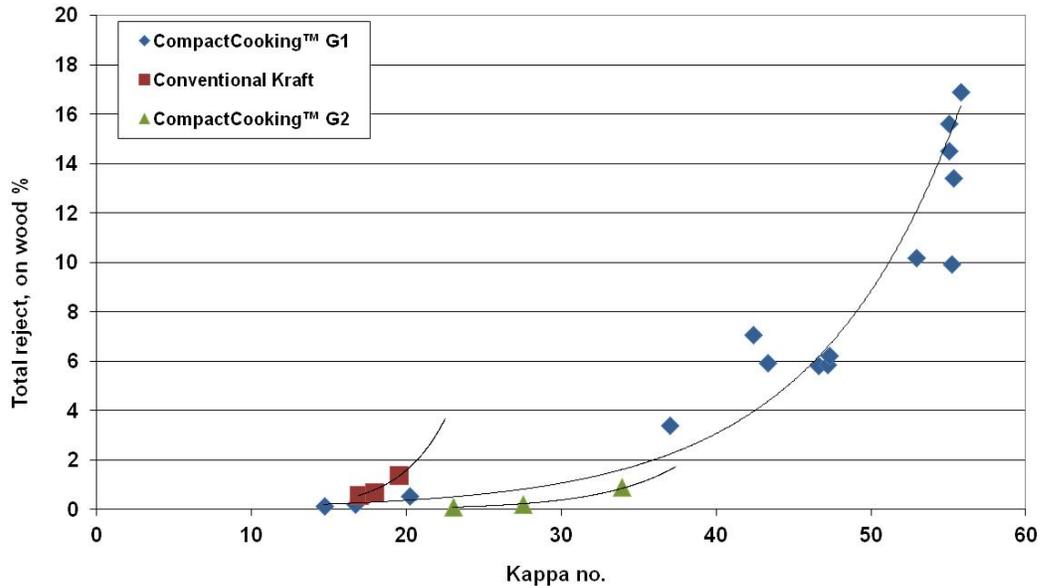
The goal of the current work was to determine conditions for a process that uses as much of the wood raw material as possible in an existing mill while minimizing the reject content of the resulting pulp. Rejects were measured as per cent on wood. A secondary goal of the work was to identify pulping conditions that would improve the strength properties of the resulting high yield pulp. The pulping process being employed utilizes both the liberated fibers obtained from the kraft cook and the reject material retained after screening (Hart et al. 2008a). Therefore, the utilization of the wood raw material is best measured by the unscreened pulp yield. If the reject portion of the cook was not utilized in the final pulp, the screened pulp yield would be a better measure of wood utilization (Hart and Connell 2006). As shown in Fig. 2, terminating the cook at higher kappa numbers increases the unscreened pulp yield. As also shown in Fig. 2, the screened pulp yield starts to decrease when the kappa number of the kraft cook is above roughly 20.

As described earlier, the reject content of the pulp as a given kappa number is influenced by the cooking process. With the CompactCooking™ concept it is possible to cook mixed Southern US hardwood chips to a kappa number of 40 to 50 with less than 15% reject on wood content (Fig. 3). Using traditional kraft cooking time/temperature profiles, the rejects content of 40 to 50 kappa number pulps are in the 25 to 30% range. By allowing good chip penetration at the beginning of the cooking process, the CompactCooking™ concept substantially increases the defibration point for the hardwood chips. In the current study, when the kappa number of the CompactCooking™ pulp was increased to about 55, a drastic increase in reject content of the pulp resulted.



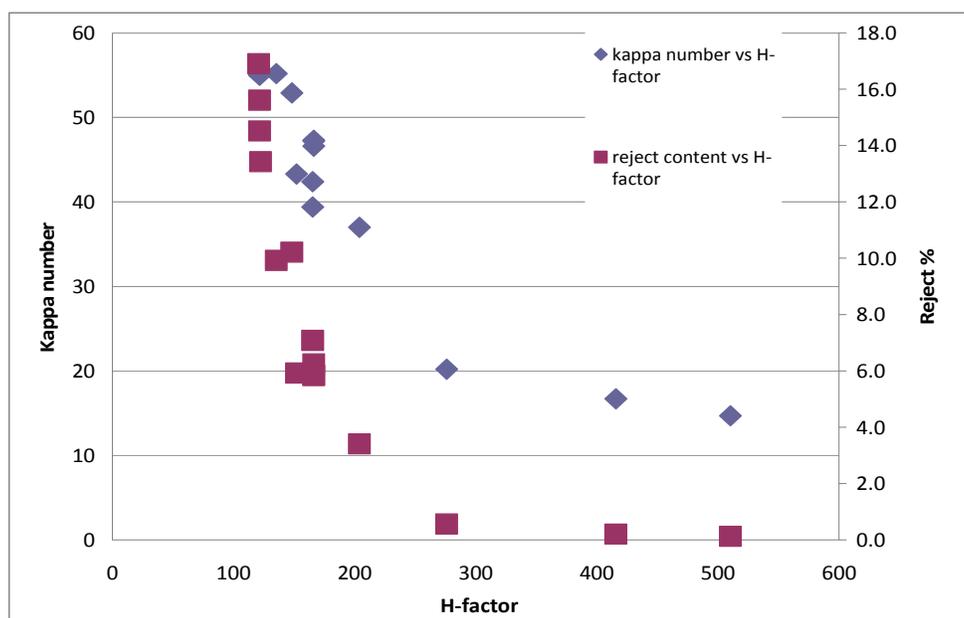
**Fig. 2.** Unscreened yield increases as function of kappa number, while the screened yield starts to decrease above a kappa number of around 20. Hence the novel pulping process utilizing both liberated fibers and rejects is necessary to benefit from an increase in kappa number. Cook time was 302 min and the temperature was varied between 266 and 193 °F (130-145°C) (The lines serve only as a guide; they do not best fit regression equations)

For the current study, the impregnation time was maintained at 18 minutes to be representative of the time available in the existing dual vessel digester available at the Evadale, TX mill. A low impregnation temperature of 125°C (275°F) was used to help enhance diffusion of active chemicals into the chips. The L:W and alkali charge were set at 5 and 22 g/L, respectively. The total alkali charge and the H-factor (cooking temperature) were varied to alter the kappa number. The kappa number and resulting rejects level are shown as a function of kappa number in Fig. 4.



**Fig. 3.** Reject content (knots and shives) on wood as function of kappa number for the Compact Cooking simulations made in this study (Compact Cooking™ G1). As comparison is data from Wedin et al. (2010) showing rejects for a conventional kraft cook and for a Compact Cooking™G2 cook performed using *Eucalyptus* as the raw material.

A set of cooks was performed to about a 43 kappa number. One cook was done with the standard 18 minute impregnation time, while the second cook had a 127 minute impregnation time. The 127 minute incorporated both the impregnation vessel and the first cooking zone residence time into a single impregnation zone with all of the cooking occurring in the second cooking zone. The residual alkali after impregnation for the longer impregnation time was considerable lower. Still, the cook with the longer impregnation time contained 1.66%-units on pulp lower rejects than the cook with the shorter impregnation time. The total percent rejects on pulp for the long impregnation time cook was 11.3% on pulp, while the total rejects of the shorter impregnation time cook was 12.9% on pulp. The shorter impregnation time cook also required an 8% increase in H-factor to obtain the same kappa number as compared to the longer impregnation time cook. As discussed above, the literature supports these findings; thus it is reasonable to assume the prolonged impregnation at lower temperature associated with the CompactCooking™ G2 process would lower the amount of rejects even further at these high kappa numbers.



**Fig. 4.** Kappa number and reject content (on wood) as a function of H-factor. As impregnation time, temperature, L:W and alkali charge are kept at 18 min, 275°F (125°C), 5,0 and 22g/L , respectively, mainly the H-factor (and the alkali charge) of the cook will determine the reject content.

### Brownstock Strength Properties

If the pulps are used in unbleached board applications, the strength of the pulp at different kappa numbers is of interest. One of the strength parameters usually measured on a pulp sheet is tensile stiffness. Tensile stiffness (Table 3) will for example, together with caliper and density of the sheet, determine the bending stiffness. Bending stiffness is especially important for packaging board grades. As the kappa number increases, the tensile stiffness of the pulp decreases slightly.

It has been shown that higher lignin content will decrease the stiffness of the sheet at a given sheet density (Antonsson et al. 2009). As the major load bearing material in pulp fiber is believed to be cellulose, it is reasonable to assume that increasing the kappa number i.e. lignin content of the fiber would decrease the tensile stiffness of the resulting pulp sheet.

In addition to tensile stiffness, other physical properties were measured on the unbeaten brown fiber sheets. Some of these properties are shown in Table 3. Over the range of kappa numbers tested, no significant difference could be determined in tear strength. As with tensile, a slight decrease in air resistance was noted with increasing kappa number. A lower air resistance might be an indication of less bonded sheets at the increasing kappa numbers, although the sheet densities were all very similar.

The current study did not separately and optimally refine the resulting rejects stream prior to physical testing. As laboratory and pilot scale trials of the new pulping process have not shown any loss of physical properties, it is probable that the increase in fiber length distribution resulting from blending the refined rejects stream with the primary accepts stream offsets any loss in fiber bonding experienced in the current study.

**Table 3.** Physical Properties of Brown Pulps at Different Kappa Numbers

Kappa No.	Sheet Density (kg/m <sup>3</sup> )	Tensile Stiffness Index (kNm/g)	Bending Stiffness Index (Nm <sup>6</sup> /kg <sup>3</sup> )	Tensile Index (Nm/g)
18.1	410	4.0	12.9	27.3
37.0	410	4.0	21.9	25.9
43.3	410	3.8	20.1	21.3
46.6	400	3.5	20.5	23.5
55.2	410	3.4	18.6	21.1

### Bleaching Study

A 46.3 kappa extended impregnation laboratory pulp (1320 ml/g viscosity), a 15.2 kappa extended impregnation laboratory pulp (1070 mL/g viscosity), and a 10.6 kappa mill hardwood pulp (1060 ml/g viscosity) were used for this study. The 46.3 kappa laboratory pulp and the 10.6 kappa mill pulp both had 14% curl before bleaching. The high kappa lab pulp was subjected to oxygen delignification. The resulting pulp kappa number was 31.8 for a kappa reduction of 31.3%. The oxygen-delignified pulp, the lower kappa lab pulp, and the mill pulp were each bleached according to D(EP)DD and XD(EP)DD sequences.

The long impregnation time, high kappa pulp showed better bleachability than the mill pulp, i.e. the high impregnation pulp required less active chlorine per kappa unit drop than the mill pulp to obtain an 89% ISO brightness. The mill pulp also had a lower final bleached viscosity than the high impregnation pulps. Data for the bleached pulps at 89% ISO brightness are located in Table 4. The use of enzymes in the bleaching sequence successfully reduced the amount of active chlorine required to obtain the target brightness. Enzyme usage also improved the final bleached pulp viscosity.

**Table 4.** Selected Data for Bleached Pulps at 89% ISO Brightness

Pulp Used	46.3 Kappa, Long Impregnation Lab Pulp			15.2 Kappa, Long Impreg Lab Pulp	10.6 Kappa Mill Pulp	
	D(EP)DD	OD(EP)DD	OXD(EP)DD	D(EP)DD	XD(EP)DD	D(EP)DD
Act Cl Consumed kg/ODT	115	76	71	54	46	48
Kappa Factor, kg act Cl/kappa	2.5	2.4	2.2	3.6	4.3	4.5
Viscosity, ml/g	1250	1020	1050	950	900	860
Reverted Bright., % ISO	86.0	86.5	86.5	86.3	86.5	86.6

Select pulps were beaten in a PFI mill and subjected to physical property testing. The physical test results were determined at 1.39 cm<sup>3</sup>/g bulk and are shown in Table 5. The high kappa, long impregnation time pulp had equivalent or better physical properties as compared to the mill pulp for every bleaching sequence examined.

**Table 5.** Selected Physical Properties of 89% ISO bleached Pulps \*

Pulp	46.3 Kappa, Long Impregnation Lab Pulp			10.6 Kappa Mill Pulp
Sequence	D(EP)DD	OD(EP)DD	OXD(EP)DD	XD(EP)DD
Tear Index, mNm <sup>2</sup> /g	10.97	10.40	10.82	9.20
Burst Index, kPam <sup>2</sup> /g	5.70	5.18	5.06	4.53
Tensile Index, Mm/g	86.2	78.1	79.9	73.0
Tensile Stiffness Index MN,/kg	7.6	7.4	7.4	7.6
TEA Index, J/Kg	2129	2010	2069	1839

\* All physical properties evaluated for pulps beaten to 1.39 cm<sup>3</sup>/g bulk

## CONCLUSIONS

1. Cooking of mixed US Southern hardwood using CompactCooking™ technology results in lower reject levels at increased kappa numbers compared to conventional cooking. Using this technology, together with a separate treatment of the reject stream, can result in improved utilization of the wood entering the mill.
2. To some extent, brownstock pulps of higher kappa number have slightly lower strength properties, which can be explained by relatively less load-carrying cellulose in the resulting unbleached fiber and decreased bonding between the fibers.
3. When these pulps are bleached to high brightness, they have as high or higher physical properties as conventional cooked, bleachable grade kappa pulps.
4. The use of enzymes in the bleaching process was found to improve the bleachability of the resulting fiber.
5. Long impregnation, high kappa pulps were found to have improved bleachability (kg ClO<sub>2</sub> consumed per kappa drop) as compared to conventional cooked, bleachable grade pulps.

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