A method of preparing dissolving pulp. The method includes physically separating a kraft pulp or a kraft hydrolysis pulp into first and second fractions, the first fraction having a relatively low lignin content and the second fraction having a relatively high lignin content.
Figure 2

Viscosity (mL/g) vs. Enzyme Dosage (u/g)

Reactivity (%) vs. Enzyme Dosage (u/g)
FIELD

This invention relates to the treatment of kraft pulp in the production of dissolving pulp.

BACKGROUND

Dissolving pulp is a raw material for manufacturing cellulose derivatives and regenerated cellulose. Production of dissolving pulp is growing. There are two commercial processes used for production of dissolving pulp. One is the relatively well known oxid sulfite process, the other being production of dissolving pulp from pulp produced via a prehydrolysis kraft process, which is relatively new. It is commonly found that adaptation of current kraft-based installations to dissolving pulp production has not been entirely successful, at least to the extent that it seems that dissolving pulps obtained to date have lower reactivity than those obtained via sulfite-based processes.

FOCK reactivity, defined as the % of cellulose dissolved under the conditions specified, is an important parameter in determining the suitability of a dissolving pulp for such purposes. Reactivity is a measure of a dissolving pulp’s ability to chemically react with, for example, carbon disulfide and sodium hydroxide in rayon production, or more specifically the ability for hydroxyl groups on the glucose units of cellulose chains to react with carbon disulfide.

Production of pulps described as suitable for lyocell manufacture has been described in the patent literature, for example, in U.S. Patent Publication No. 2009/0165969.

SUMMARY

In one aspect, the invention is a method of preparing dissolving pulp, the method comprising the steps of:

(i) separating a kraft pulp into a fiber fraction and a fines fraction; and
(ii) processing the fiber fraction, to produce a dissolving pulp.

The separation step is carried out so as to obtain a first fraction having a relatively low lignin content and a second fraction having a relatively high lignin content. In certain embodiments, the separation step includes separating specific particle fractions on the basis of size, wherein particles of the first fraction are relatively large and particles of the second fraction are relatively small. The second fraction can have a lignin content of at least about 15% by weight, or 16%, of 17%, or 18%, or 19%, or 20%, or 21%, or 22%, or 23%, or at least 24%. The first fraction can have a lignin content of no more than about 1%, or 2%, or 3%, or 4%, or 5%, or 6%, or 7%, or 8%, or 9%, or 10%, or 11%, or 12%, or 13%, or no more than about 14%.

In one aspect, the kraft pulp is subjected to a step (i) thus separated into the fiber and fines fractions according to size to produce a fines fraction having a lignin content higher than the lignin content of the fiber fraction.

In embodiments, the unseparated kraft pulp has a kappa number of between 10 and 50.

The kraft pulp subject of separating step (i) can have a kappa number of between 10 and 40.

The kappa number of the first fraction can be no more than about 80% the kappa number of the unseparated pulp.

The method can be used with kraft pulp produced by a conventional kraft pulping process, or the kraft pulp can be a prehydrolysis kraft pulp. In the latter case, the kraft pulp would generally have a kappa number of no more than about 20. The pulp may be produced as a separate stock, and processed separately, e.g., in a separate mill, or kraft pulping wood chips to produce the kraft pulp may be carried out in-line along with subsequent steps to produce the dissolving pulp. The wood chips can be subjected to steam/water prehydrolysis prior to the kraft pulping.

The method can thus include a step of, prior to step (i), kraft pulping wood chips to produce the pulp. Wood chips can include softwood and the wood chips can be kraft pulped to obtain a pulp having a kappa number of, e.g., less than 40, or between 20 and 40, or between 25 and 40, or between 30 and 40. Softwood chips can include, e.g., spruce, hemlock, fir, larch, or combinations of any of the foregoing. Hardwood chips can be kraft pulped to obtain a pulp having a kappa number of less than 20, or between 10 and 20, or between 10 and 15. Hardwood chips can include, e.g., maple, aspen, birch, or combinations of any of the foregoing.

According to certain embodiments, wood chips are subjected to steam and/or water prehydrolysis prior to kraft pulping. The kraft pulp can be obtained from a conventional kraft process without a prehydrolysis step, and the pulp subject of separating step (i) can have a kappa number of between 10 and 40, or between 30 and 40.

In embodiments, the lignin content (wt %) of the second fraction is at least 1.1 the lignin content of the first fraction, more preferably at least 1.2, 1.3, 1.4 or 1.5 of the first fraction. Further, between 85 and 95 wt % of the unfractionated kraft pulp can be obtained in the first fraction obtained in step (i).

The method can further include a step (iii), of exposing the fiber fraction to a cellulase, xylanase, and/or mannanase enzyme to decrease the intrinsic viscosity of the pulp fraction by between 50 and 700 mll/g (0.5% cellulose in a cupriethylenediamine (CED) solution). More preferably, the decrease is in the range of from 100 to 300 mll/g. The cellulase enzyme can be a multicomponent enzyme or an enzyme having a single type of catalytic activity. FiberCare D™ can be used in a dosage of 0.1 to 10 ug of dry pulp, preferably, 0.3 to 2 ug of pulp. Examples of other commercially available enzymes are those sold under the names FiberCare U, FiberCare R, Celaclast, Fiberzyme CS, Fiberzyme G200, Fiberzyme LBR, Optimar CX, Pyrolase HT cellulose, Pulzyme HB, Pulzyme HC, Laminase PB-100 and Mannaway®. These can be used alone or in combination with each other.

The method can include exposing the pulp fraction to one or more cationic polymers during such an enzyme treatment step. Cationic polymers can be included in enzymatic steps, particularly cellulase-catalyzed steps to enhance enzymatic performance. The polymer can be, but are not limited to a cationic polyacrylamide (CPAM), polydiallyldimethylammonium chloride (DADMAC), polyethyleneimine (PEI), polyaluminum chloride (PAC), cationic starch, or a dual polymer system, and the cationic polymer or polymers can further include an anionic polymer, microparticle-containing system, and/or a silica-based cationic polymers. If present, polymer is present at e.g., a dosage in the range of 0.01 to 1000 ppm.

In embodiment the method includes a step of (iv) bleaching the first pulp fraction before, after or as part of step (iii). The method can further include a step of (v) exposing the
bleached pulp obtained in step (iv) to a xylanase. Such method can further include the step of (vi) subjecting the pulp obtained in step (v) to alkali.

**[0018]** Step (vi) can be a hot alkali treatment, or step (vi) can be a cold alkali treatment with the caustic soda concentration in the range of 1 to 12 wt %, preferably, 5 to 12 wt %, even more preferably 8 to 10 wt %.

**[0019]** In embodiments that include enzymatic treatment step (iii), the method can further include recovering active enzyme of step (iii) and cycling the recovered enzyme back into step (iii). The amount of enzyme recycled can be in the range of 30 to 90%, more preferably 50 to 80%. In embodiments that include step (v), the method can include recovering active enzyme of step (v) and cycling the recovered enzyme back into step (v).

**[0020]** The dissolving pulp produced typically has a relatively low lignin content of no more than about 0.3 wt %, no more than about 0.2 wt %, or no more than about 0.1 wt %.

**[0021]** The dissolving pulp produced typically has an α-cellulose content of at least about 88 wt %, at least about 90 wt %, or at least about 92 wt %, or at least about 94 wt %, or at least about 96%, or higher.

**[0022]** The dissolving pulp produced typically has a pentosan content of no more than about 8 wt %, or less than about 7 wt %, or less than about 6 wt %, or less than about 5 wt %, or less than about 4 wt %.

**[0023]** The pulp of step (i) can be unbleached pulp.

**[0024]** The separating step is carried out by, e.g., centrifugal separation, such as by use of a hydroclone or series of screens, or similar setups, or it can be by screening of the kraft pulp into the fiber fraction and fines fraction.

**[0025]** The method can include (iv) bleaching the pulp, before, after or as part of step (iii). The method can further include the step of (v) exposing the bleached pulp to a xylanase. The method can also include the step of (vi) subjecting the pulp produced in step (v) to alkali. Step (vi) can be a hot alkali treatment or a cold alkali treatment, as discussed further below.

**[0026]** The method can also include a step of refining the fiber fraction to produce the dissolving pulp. The Fock reactivity of the dissolving pulp produced can be at least 20% more preferably, 30%, 40%, 50%, more preferably at least 60%.

**[0027]** The method can also include a step of (a) exposing the second i.e. fines fraction to a cellulose enzyme, mechanical and/or alkali treatment.

**[0028]** The method can further include a step of (b) bleaching the fines fraction i.e., step (a) can be carried out prior to, subsequent to, or simultaneously with step (b). Such steps can be carried out in different orders, and can be repeated.

**[0029]** It can be possible to process the fines fraction separately to produce a pulp that might be used as stock to produce a paper product, or the fines could be, or at least a portion of the fines could be, processed into a dissolving pulp and rejoined with the fiber fraction.

**BRIEF DESCRIPTION OF THE DRAWINGS**

**[0030]** FIG. 1 shows a flow diagram incorporating process steps of the present invention; and

**[0031]** FIG. 2 shows Fock reactivity and viscosity of enzyme treated dissolving pulps produced from the prehydrolysis kraft-based process from a mixture of hardwood. Enzymatic treatment was performed at pH 5.0 and temperature 55° C. for 2 hours.

**[0032]** FIG. 3 shows the effect of the addition of 250 ppm CPAM on the change in viscosity over time of pulp (consistency 3%, pH 4.8 and 55° C.) treated with cellulase (mg/g pulp): 0.5 (■), 1 (▲), and 1.5 (▲). Cellulase charge (mg/g pulp) with no added CPAM: 0.5 (□), 1 (○) and 2 (□) (hollow inverted triangles).

**[0033]** FIG. 4 shows Fock reactivity of pulp (3% pulp consistency, pH 4.8 and 55° C.) after 12 hours (hatched bars) and 24 hours (unhatched bars) of cellulase treatment. The first, second and third pairs of bars are 0.5, 1 and 2 mg of cellulase charge per g of pulp with no added CPAM. The second, fourth and sixth pairs of bars are 0.5, 1, and 1.5 mg of cellulase charge per g of pulp and 250 ppm CPAM.

**DETAILED DESCRIPTION**

**[0034]** This invention discloses a cost effective and environmentally friendly method for improving the product quality parameters and processing efficiency in the production of dissolving pulp by the prehydrolysis kraft process or kraft process.

**[0035]** Pulp quality, such as Fock reactivity, purity e.g., high α-cellulose and low pentosan content, accessibility, is important for dissolving grade pulp, these quality parameters being important in the downstream production process regardless of the pathways of functionalizing cellulose during viscose production and processing.

**[0036]** Dissolving pulp from a prehydrolysis kraft pulp process or from the kraft pulping process contains undesirable carbohydrates components, including residual hemicelluloses after cooking, such as glucuronomannan, glucuronoxylan. In addition, some degraded cellulose chains might be too short or altered to suit the properties of certain products such as cord rayon, high tenacity staple fiber, modal fiber, and should therefore be removed. Portions of the pulp that are smaller in size, such as ray cells and fines, have inferior properties compared to those that are larger in size, such as tracheid.

**[0037]** According to the present invention, the dissolving pulp quality e.g., purity of the alpha-cellulose, reduced pentosan content, increased reactivity and accessibility, can be improved cost-effectively by additional purification steps or combining the purification stages/stages with the existing manufacturing process, especially in brown stock operations and bleach plant operations. The purification of dissolving pulp can be conducted in a controllable manner to meet the end-use specifications.

**[0038]** As shown in FIG. 1, a fractionation stage in which a rejects portion containing relatively large proportions of fines and ray cells is separated from fibers after prehydrolysis and kraft pulping can include additional steps such as subsequent bleaching, enzyme treatment and/or alkali purification.

**Fractionation**

**[0039]** U.S. Pat. No. 4,731,160 describes the fractionation method for mechanical pulp into a major fraction and fines fraction. U.S. Pat. No. 7,005,034 discloses a method for production of mechanical pulp, wherein the pulp is fractionated after a first refinery stage for separating the fine material from the pulp to facilitate the refining of the fibers in the second stage. U.S. Patent Publication No. 2007/0023329 describes a method for selective removal of ray cells from pulp by combining screening and centrifugal cleaning.
Unknown to the inventors is the description in literature of the fractionation of a pulp to be used as, or in the production of, a dissolving pulp, either from the prehydrolysis kraft pulping process or the kraft process in order to obtain a fraction containing a lowered lignin content.

According to the present invention, unbleached pulp from the kraft pulping stage contains a small percentage of ray cells and fragments of fibers and middle lamellae. This portion of pulp is called fines. The fines portion has significantly higher content of impurities such as lignin, resin, extractives, and non-process elements like iron, calcium, and magnesium. In addition, the fines portion has a much slower drainage rate, and inferior bleach-ability resulting in higher bleach chemicals consumption and accessibility. Although the fines account for only a small amount (usually less than 8%) of the mass of the whole pulp, their negative impact on the performance and efficiency of the bleaching, enzymatic treatment and alkali purification stages, as well as the overall quality of the final dissolving pulp, is significant.

According to the invention, unbleached pulp is separated by fractionation into two fractions: a fiber fraction and a fines fraction, which are separately processed. This leads to overall chemical and energy efficiencies and/or improved quality parameters of dissolving grade pulp ultimately obtained from the fiber fraction. FIG. 1 illustrates an embodiment of the invention.

Dissolving pulp can be produced in a more cost-effective manner by fractionating pulp first to improve the chemical response and drainage characteristics of the majority of the dissolving grade pulp (the fiber fraction) in the bleaching, enzymatic treatment and/or alkali purification step and/or mechanical refining of fibers. The fines fraction separated by the fractionation step, can be processed by bleaching, alkali extraction and enzymatic treatment.

It thus becomes possible for regular kraft production to be the basis for production of dissolving grade pulp suitable for viscose process (xanthation), or for other cellulose derivatives production.

The process can be varied based on the desired purity of pulp.

Unbleached pulp from the kraft pulping stage is separated into a minor fines fraction and major fiber fraction. The separation may be accomplished using a screen, or centrifugal separators (cyclones). The major fiber fraction is typically subjected to enzymatic treatment and then subsequent bleaching sequences to produce fully bleached pulp having higher brightness and whiteness than pulp bleached without a separation stage at the same chemical dosage, or to produce dissolving pulp at lower chemical dosage and lower energy consumption for given pulp brightness and whiteness.

After bleaching, the fiber fraction is subjected to enzymatic treatment and/or cold alkali purification and/or refining to increase the cell-wall cellulose content to the specific target, for example, about 98%, and increase Fock reactivity, in a more efficient way in terms of energy and chemical consumption when compared to a similar process that does not include such a fractionation stage. Table 1 shows the mass percentage of lignin content for different pulp fractions of one kraft brown stock pulp.

As shown in Table 1, the lignin content is much different in different fractions of the brown stock pulp, the longest fraction (>50 mesh size) accounts for about 90% of the mass, but the lignin content is the lowest (5.5%). One can thus see that (i) residual lignin in unbleached pulp is unevenly distributed, and (ii) the smallest fine fraction (mostly ray cells) contains much higher lignin content than the fiber tracheids (long fibers). If the fines fractions would be separated from the tracheids, due to the significantly lower lignin content, smaller amounts of bleaching chemicals would be needed in the subsequent bleach plant. Also, if the fines are removed, the filterability of the dissolving pulp should be improved.

As the separation processes described herein are, ultimately, toward the production of a dissolving pulp, the pulp subject of the separation, fractionation or screening process has a suitably low overall lignin content. This is reflected in the kappa number of the pulp prior to the separation step. For conventional softwood kraft pulp, the kappa number may be in a range of 30 to 40; for a prehydrolysis kraft-based dissolving pulp, in particular, hardwood pulp, the kappa number can be substantially lower, in the range of 10 to 16, as lignin is generally considered an undesirable component of dissolving pulps. The results shown in Table 1 were obtained using a conventional softwood kraft pulp having a kappa number of about 38.

The fines fraction can be treated separately, and also more efficiently by the processes involved, since the volume of the fraction is smaller than that of the long fiber fraction. As shown in Table 1, the mass percentage of the fines fraction (passed 200 mesh) is about 5%, but its lignin content is several times that of the fiber fraction (fibers retained on 100 mesh or longer). Table 2 shows lignin and metal content of pulp fibers and ray cells, the pulp being without any fractionation, and the ray cells being obtained by screening. As can be seen, the ray cells, a major component of the fines fraction, have a much higher iron and other metal content than the pulp as a whole, so fines separation by fractionation would decrease iron content of the pulp.

The fines fraction can thus be subjected to an acid treatment stage to remove the iron more efficiently, and then bleached by a bleaching sequence (e.g. D2E4O6H2O2Fe2+D2: D0 chlorine dioxide pre-bleaching, E4O6: oxygen and peroxide re-enforced alkali extraction, H2: hypochlorite, E2O5 peroxide re-enforced extraction, D2: second chlorine dioxide brightening stage). The fines fraction may also be treated by the enzyme treatment and alkali purification if needed, again in a more efficient way.

In tests it was found that removal of the fines leads to the dissolving pulp having improved drainage properties. This is shown in Table 3 by the increase of the filterability (or CSF freeness) of the pulp that was 60% maple, 30% aspen and 10% birch, all of which are hardwoods. In addition, as shown in Table 3, the brightness is much higher for the long fiber
fraction than the shorter fiber fraction, while S1,X extractives, and pentosan for the long fiber fraction, are lower.

**TABLE 2**

<table>
<thead>
<tr>
<th>Lignin [%]</th>
<th>Mn [mg/L]</th>
<th>Cu [mg/L]</th>
<th>Fe [mg/L]</th>
<th>Mg [mg/L]</th>
<th>Ca [mg/L]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pulp</td>
<td>3.38</td>
<td>100</td>
<td>1.2</td>
<td>12</td>
<td>270</td>
</tr>
<tr>
<td>Ray Cell</td>
<td>8.1</td>
<td>178</td>
<td>25</td>
<td>146</td>
<td>587</td>
</tr>
</tbody>
</table>

**TABLE 3**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Feed (&gt;100 mesh)</th>
<th>Short fibers (&lt;200 mesh)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S10</td>
<td>%</td>
<td>6.5</td>
<td>5.6</td>
</tr>
<tr>
<td>S20</td>
<td>%</td>
<td>4.2</td>
<td>3.5</td>
</tr>
<tr>
<td>Extractive</td>
<td>%</td>
<td>0.15</td>
<td>0.10</td>
</tr>
<tr>
<td>Ash</td>
<td>%</td>
<td>0.04</td>
<td>0.02</td>
</tr>
<tr>
<td>Pentosan</td>
<td>%</td>
<td>2.88</td>
<td>2.8</td>
</tr>
<tr>
<td>Brightness</td>
<td>%</td>
<td>89</td>
<td>90.5</td>
</tr>
<tr>
<td>Filterability</td>
<td>ISO</td>
<td>428</td>
<td>428</td>
</tr>
</tbody>
</table>

**Enzymatic Treatment**

[0052] In a prehydrolysis kraft process, as in the example of pulp obtained here from the AV Nackawic mill, the majority of the hemicelluloses in wood chips is degraded in the acidic prehydrolysis step and dissolved in the subsequent Kraft cooking stage. Typically, unbleached pulp from the cooking process is bleached to about 98% ISO brightness by a five-stage bleaching sequence (for example, D3,C3OPH2O,D2), chlorine dioxide pre-bleaching, E2, oxygen and peroxide re-enforced alkali extraction, H5: hypochlorite, E3, peroxide re-enforced extraction, D2: second chlorine dioxide brightening stage). Most of the lignin and wood extractives are also removed in the cooking and bleaching processes. Upon drying, the remaining cellulose fibrils are able to bind more closely to each other to form a compact structure. The inter fibrillar spaces of the pulp fibers collapse and thus the surface area and pore volume decrease [2-5]. This irreversible process is called hornification which causes decreased pulp accessibility/reactivity [6,7]. The method of drying influences the size of the fibril aggregate. Harsher drying (fast drying) produces larger lateral fibril aggregates, resulting in lower reactivity [8]. The hornification effect is more pronounced in the presence of hemicelluloses that has a very high bonding capability. This is one of the reasons why the hemicellulose content of dissolving pulp has to be reduced to a very low level.

[0053] Enzyme hydrolysis/ degradation and chemical modification have been proposed to increase the accessibility/reactivity of dissolving pulp [5,9]. Within the past few years, a monocomponent endoglucanase has been used to treat dissolving pulp to increase accessibility/reactivity with promising results [10,11]. Pure monocomponent cellulases of the endoglucanase type are commercially available.

[0054] The activation mechanism of dissolving pulp by endoglucanases is not completely understood, although hypotheses have been put forward [12,13]. It is generally believed that the attack on the less ordered cellulose regions by the endoglucanase leads to fiber wall swelling and thus an increase in accessibility towards solvents and reactants. Endoglucanase preferably degrades amorphous cellulose located on the fiber surface and between the microfibrils, which leads to increased crystalline surface exposure and to increased swelling ability and reactivity of the pulp [14]. Endoglucanase may also increase reactivity by attacking cellulose II [15,16]. Recent studies by Ibarra et al. reveal that the activation effect of endoglucanase is affected by the modular structure of the enzyme and the drying history of dissolving pulp [17,18]. They found that endoglucanase with an inverting catalytic domain and a cellulose binding domain is most effective in activating cellulose, in particular when it is applied on never-dried pulps.

[0055] Most studies, however, have focused on sulfite dissolving pulp, and little has been reported in the literature on the effect of enzyme treatment on the accessibility/reactivity of the dissolving pulp from a prehydrolysis kraft process. The action of the endoglucanase on kraft pulp differs from that on sulfite dissolving pulp. For example, the reactivity of a sulfite dissolving pulp increased rapidly from about 70% to almost 100% within 10 min of incubation [9]. In contrast, post endoglucanase treatment of a Kraft pulp increased its reactivity only marginally (from 19.1% to 22.9%) [19].

[0056] Endoglucanase may act on kraft pulp by a different mechanism, or it may be that sulfite and kraft pulps differ in chemical and physical properties. Modification of the bleaching process of prehydrolysis kraft pulp production may improve its Fock reactivity, as well as its response to enzyme treatment for reactivity improvement. In a recent study, a spruce prehydrolyzed kraft pulp was bleached with chlorite to remove the remaining lignin, in order to study the effect of residual lignin on the Fock reactivity [20]. The results showed that the Fock reactivity of the pulp increased with decreasing kappa number (decreasing residual lignin content), and the highest reactivity was obtained after complete lignin removal using chlorite delignification. It was also found that the carbohydrate composition had little influence on the pulp reactivity, but lower intrinsic viscosity either obtained by prolonged cooking or chlorite delignification correlated with higher pulp reactivity.

[0057] It has been found here that treating the pulp with xylanases and/or cellulases can significantly improve the purity and Fock reactivity of the dissolving pulp. Xylanases can have an effect on pore structure as well as pentosan content. The pre-bleach application is the standard application point but xylanase can be effectively applied at any point of the bleaching process where conditions are acceptable. Hemicellulose (such as xylanase) is an effective means to increase brightness and remove residual pentosan after the bleaching.

[0058] Accessibility to the substrate can limit the effectiveness of enzyme treatments, and especially so for dissolving grade pulps. For this reason, it is most effective to have complementary activities working simultaneously. Cellulases and hemicellulases can complement each other and achieve greater results than either alone. It is not always feasible to remove most of a single component without disrupting other substrate structures. Multiple applications of a particular treatment can be more effective than a single treatment at the same net dose. Pentosan removal is more efficient when a series of enzyme treatments are carried out at different points in the process, especially when there is a wash step or a different reactive step in between. This leads to the prospect of a brown stock treatment and a post bleach treatment with hemicellulases.
According to the present invention, the Fock reactivity of dissolving pulp can increase from the enzyme treatment, as shown in FIG. 2 (the reactivity and viscosity of resulting dissolving pulp as a function of the cellulase (a multi-component cellulase sample, FiberCare D, from Novozymes) dosage). It is evident that the reactivity increased from 48.0% to 93.5% when increasing the cellulase dosage from 0 to 2 μg enzyme ml⁻¹ dry pulp, and the viscosity decreased from 665.8 ml g⁻¹ to 354.7 ml g⁻¹; a further increase in the cellulase dosage only resulted in a slight increase in the Fock reactivity.

Other enzymes, such as FiberCare U, FiberCare R, Celluclast, Fiberszyme CS, Fiberszyme G200, Fiberszyme LBR, Optimase Cx, Pyrolase HT cellulose, may also be used for this purpose.

As shown in FIG. 2, the decrease in pulp viscosity corresponds roughly with the observed increase in the reactivity of dissolving pulp.

One or more cationic polymers, such as CPAM (cationic polyacrylamide) can be included in the cellulase treatment solution to enhance the performance of the cellulase treatment step. FIG. 3 shows the effect on viscosity of the inclusion of CPAM at a concentration of 250 ppm in the cellulase enzymatic treatment of dissolving pulp for different enzyme concentrations. It can be seen that the decrease in intrinsic viscosity over time is greater in the presence of CPAM for a given charge of enzyme. The viscosity of cellulase charge of 1 mg g⁻¹ from the CPAM addition was lower than that of 2 mg g⁻¹ cellulase charge without CPAM, for example, demonstrating that the cellulase efficiency was significantly improved due to the addition of CPAM.

The use of CPAM in the cellulase treatment can also enhance the increase in Fock reactivity, as shown in FIG. 4. As evident from the results shown, treating samples with cellulase-CPAM compositions resulted in pulp compositions have significantly higher Fock reactivity.

According to present invention, the pentosan content of the pulp can be decreased significantly with the enzyme treatment, Ecopulp TX-800 (an industrial enzyme product) as shown in Table 4. The yield losses for pulp treated by enzyme are from 1 to 1.3% per 1% increase in α-cellulose content.

Other enzymes may also be used, including Pulzyme 11B, Pulzyme 11C, Luminase PB-100, and Manaway®.

**TABLE 4**

<table>
<thead>
<tr>
<th>Enzyme</th>
<th>Addition (L/T)</th>
<th>pH</th>
<th>Consistency</th>
<th>Temp.</th>
<th>Retention Time</th>
<th>Washed</th>
<th>K#</th>
<th>Pentosan</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ecopulp TX-800 A*</td>
<td>0</td>
<td>7.4</td>
<td>14.23%</td>
<td>60°C</td>
<td>30 min</td>
<td>Yes***</td>
<td>5.6</td>
<td>3.56</td>
</tr>
<tr>
<td>Ecopulp TX-800 A*</td>
<td>0.04**</td>
<td>7.4</td>
<td>14.23%</td>
<td>60°C</td>
<td>30 min</td>
<td>Yes***</td>
<td>5.5</td>
<td>3.48</td>
</tr>
<tr>
<td>Ecopulp TX-800 A*</td>
<td>0.08**</td>
<td>7.4</td>
<td>14.23%</td>
<td>60°C</td>
<td>30 min</td>
<td>Yes***</td>
<td>5.4</td>
<td>3.04</td>
</tr>
<tr>
<td>Ecopulp TX-800 A*</td>
<td>0.10**</td>
<td>7.4</td>
<td>14.23%</td>
<td>60°C</td>
<td>30 min</td>
<td>Yes***</td>
<td>5.2</td>
<td>2.2</td>
</tr>
</tbody>
</table>

Conditions: 9 wt % NaOH concentration, 9 wt % pulp consistency, 30 minutes, 35°C. Each sample was washed with deionized water until pH 6-7 then air dried.

Cold Alkali Treatment

Alkaline treatment/purification of pulp can principally be carried out in two ways, cold and hot alkaline purification:

a) Cold purification consisting of the treatment of pulp in concentrated lye around room temperature, permitting short chain material and microfibril fragments to dissolve. Cold purification thus primarily involves physical changes to the pulp, and only a small amount of alkali is consumed; and

b) Hot alkali purification, performed at a higher temperature (usually higher than 70°C), and in some cases, higher than 100°C, with a relatively lower alkali dosage (0.2-4 wt % on pulp). The more accessible parts of the fiber react under these conditions, with the formation of organic acids.

**TABLE 5**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Reactivity (%)</th>
<th>Viscosity (ml/g)</th>
<th>Pentosan (%)</th>
<th>α-Cellulose (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before alkaline treatment</td>
<td>39.2</td>
<td>605.0</td>
<td>4.42</td>
<td>94.3</td>
</tr>
<tr>
<td>After alkaline treatment</td>
<td>20.8</td>
<td>641.6</td>
<td>1.41</td>
<td>97.9</td>
</tr>
</tbody>
</table>

Conditions: 9 wt % NaOH concentration, 9 wt % pulp consistency, 30 minutes, 35°C. Each sample was washed with deionized water until pH 6-7 then air dried.

Samples were tested 24 days after treatment.

* Diluted 1 ml/100 ml.
** Additives: 0.4 ml/g 0.8 ml/10 g, 1.0 ml/10 g of diluted enzyme.
*** Washed in sheet former with 15 L of water purified by reverse osmosis; dried in oven.

BOD Used the 4LT sample 1 ml enzyme diluted to 1000 ml 0.4 ml of the enzyme diluted to 2000 ml = 0.000002 ml of enzyme that sample gave 12 mg/l BOD.
Suitable ranges for the temperature and alkali concentration for the cold alkalipurification are 5 to 40°C and 5 to 18%. Under these conditions, mainly physical changes occur. 35°C is a preferred temperature for the prehydrolysis kraft dissolving pulp. Cold alkali treated pulp is sensitive to oxidative attack, which may lead to a decrease in the pulp viscosity. Reaction time can be short and the swelling reaction takes place almost instantaneously. Pulp consistency can vary from 3.5 to 20%. The pentosan content of the pulp decreases as the α-cellulose content is increased after the cold alkali treatment.

**Hot Alkali Purification**

In contrast to cold purification, the hot alkali treatment involves chemical reactions in the purification process. Swelling is limited, as the concentration of lye in the reaction mixture is only 0.2-4 wt % NaOH. The degree of purification is regulated by the alkali concentration, as well as by the temperature, time and pulp consistency. In comparison to the cold alkali treatment, the main drawbacks of the hot alkali method are the consumption of steam for heating the pulp and higher yield loss of pulp. A yield loss of about 3% is expected per 1% increase in α-cellulose content.

**Mechanical Treatment**

Mechanical treatment of pulp fibers, such as grinding, refining, can improve the dissolving pulp reactivity. Fock reactivity was determined at 18°C, by dissolving pulp sample ground in a coffee grinder, i.e., for refining/grinding. As indicated by the results shown in Table 6, Fock reactivity increases with grinding time. Fiber length was determined by FQA.

<table>
<thead>
<tr>
<th>No.</th>
<th>Grinding Time</th>
<th>Fiber Length (length weighted)</th>
<th>Fock Reactivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0 min</td>
<td>0.545 mm</td>
<td>55.90%</td>
</tr>
<tr>
<td>2</td>
<td>0.5 min</td>
<td>0.536 mm</td>
<td>59.10%</td>
</tr>
<tr>
<td>3</td>
<td>1 min</td>
<td>0.514 mm</td>
<td>61.08%</td>
</tr>
<tr>
<td>4</td>
<td>3 min</td>
<td>0.486 mm</td>
<td>65.56%</td>
</tr>
<tr>
<td>5</td>
<td>5 min</td>
<td>0.466 mm</td>
<td>68.67%</td>
</tr>
<tr>
<td>6</td>
<td>10 min</td>
<td>0.433 mm</td>
<td>75.13%</td>
</tr>
</tbody>
</table>

**Enzyme Recycling**

Enzyme recycling relates to the process economics of the enzyme treatment. After use, the filtrate containing active enzymes and can be recycled/reused. Table 7 shows effects of recycle of filtrate from enzymatic treatment on dissolving pulp reactivity.

**REFERENCES**


The method of claim 7, wherein said wood chips are subjected to steam and/or water prehydrolysis prior to said kraft pulping.

9. The method of claim 7, wherein the kraft pulp is obtained from a conventional kraft process without a prehydrolysis step, and the pulp subject of separating step (i) has a kappa number of between 10 and 40.

10. The method of claim 9, wherein the lignin content (wt %) of the second fraction is at least 11.1 the lignin content of the first fraction.

11. The method of claim 10, wherein between 85 and 95 wt % of the unfractionated kraft pulp is contained in the first fraction obtained in step (i).

12. The method of claim 11, further comprising the step of (iii) exposing the first pulp fraction to a cellulase, xylanase, and/or mannanase enzyme to decrease the intrinsic viscosity of the pulp fraction by between 50 and 700 ml/g (0.5% cellulose in a cupriethylenediamine (CED) solution), optionally exposing the pulp fraction to one or more cationic polymers wherein said polymer can be selected from the group consisting of cationic polyacrylamide (CPAM), polydiallyldimethylammonium chloride (DADMAC), polyethyleneimine (PEI), polyaluminum chloride (PAC), cationic starch, a dual polymer system, wherein the cationic polymer can further comprise an amionic polymer, microparticle-containing system, and/or a silica-based cationic polymers.

13. The method of claim 12, wherein the enzyme comprises FiberCare D™ present in a dosage 0.1 to 10 mg of dry pulp; further comprising the step of (iv) bleaching the first pulp fraction before, after or as part of step (iii); further comprising the step of (v) exposing the bleached pulp obtained in step (iv) to a xylanase; further comprising the step of (vi) subjecting the pulp obtained in step (v) to alkalization; and wherein step (vi) comprises a hot alkalization treatment, or step (vii) comprises a cold alkalization treatment and the caustic soda concentration is in the range of 1 to 12 wt %.

14. The method of claim 13, further comprising recovering active enzyme of step (ii) and cycling the recovered enzyme back into step (iii), wherein amount of enzyme recycled is in the range of 30 to 90%.

15. The method of claim 6, wherein step (i) comprises centrifugal separation.

16. The method of claim 6, wherein step (i) comprises screening of the kraft pulp into the first and second fractions.

17. The method of claim 6, wherein the kraft pulp of step (i) is a prehydrolysis kraft pulp.

18. The method of claim 6, further comprising the step of refining the fiber fraction to produce the dissolving pulp.

19. The method of claim 18, wherein the dissolving pulp produced has a lignin content of no more than about 0.3 wt %.

20. The method of claim 19, wherein the dissolving pulp produced has an a-cellulose content of at least about 88 wt %.

21. The method of claim 20, wherein the dissolving pulp produced has a pentosan content of no more than about 8 wt %.

22. The method of claim 21, wherein the Fock reactivity of the dissolving pulp produced is at least 50%.

* * *