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(54) Use of wash press for pulp alkali addition process.

 $\bigcirc$  Unbleached pulp is combined with an aqueous alkaline solution while in a state of low consistency (9) to distribute a first amount of alkaline material substantially uniformly throughout the pulp. The consistency of the pulp is then increased (12) to above about 18%, and the high consistency alkali containing pulp is then treated with oxygen (21) to effect delignification (20). Additional alkali may be applied onto the high consistency pulp prior to oxygen delignification to provide a total amount of between 0.8 and 7% by weight of oven dry pulp. The invention contemplates the use of one or more wash presses (7,30) for reducing the overall amounts of alkaline material utilized in the process. High strength, low lignin pulps (25) are subsequently formed which may be further bleached to high brightness with reduced amounts of chemicals by following the methods of the invention.



#### FIELD OF INVENTION

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The present invention relates to a method for the treatment of wood pulp, and more particularly to a method for oxygen delignification of brownstock to produce highly delignified pulp without deleteriously affecting strength.

# BACKGROUND OF THE INVENTION

Wood is comprised in major proportion of cellulose and hemicellulose fiber and amorphous, non-fibrous lignin which serves to hold the fibrous portions together. The hemicellulose and the cellulose are sometimes 10 referred to collectively as holocellulose. During the treatment of wood to produce pulp, the wood is transformed into a fibrous mass by removing a substantial portion of the lignin from the wood. Thus, processes for the production of paper and paper products generally include a pulping stage in which wood, usually in the form of wood chips, is reduced to a fibrous mass. Several different pulping methods are known in the art; they are generally classified as mechanical, chemical or semi-chemical pulping. 15

Chemical pulping methods include a wide variety of processes, such as the sulfite process, the bisulfite process, the soda process and the Kraft process. The Kraft process is the predominant form of chemical pulping.

Chemical pulping operations generally comprise introducing wood chips into a digesting vessel where they are cooked in a chemical liquor. In the Kraft process, the cooking liquor comprises a mixture of sodium 20 hydroxide and sodium sulfide. After the required cooking period, softened and delignified wood chips are separated from the cooking liquor to produce a fibrous mass of pulp. The pulp produced by chemical pulping is called "brownstock." The brownstock is typically washed to remove cooking liquor and then processed for the production of unbleached grades of paper products or, alternatively, bleached for the production of high brightness paper products.

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Since chromophoric groups on the lignin are principally responsible for color in the pulp, most methods for the bleaching of brownstock require further delignification of the brownstock. For example, the brownstock may be reacted with elemental chlorine in an acidic medium or with hypochlorite in an alkaline solution to effect this further delignification. These steps are typically followed by reactions with chlorine

- dioxide to produce a fully bleached product. Oxygen delignification is a method that has been used at an 30 increasing rate in recent years for the bleaching of pulp because it uses inexpensive bleach chemicals and produces by-products which can be burned in a recovery boiler reducing environmental pollutants. Oxygen delignification is frequently followed by bleach stages which use chlorine or chlorine dioxide but require less bleach chemical and produce less environmental pollutants because of the bleaching achieved in the oxygen stage. 35

In some bleaching processes, the pulp is bleached while being maintained at low to medium levels of pulp consistency. Pulp consistency is a measure of the percentage of solid fibrous material in pulp. Pulps having a consistency of less than about 10% by weight are said to be in the low to medium range of pulp consistency. Processes which require bleaching at low to medium consistency are described in the

- following patents and publications: U.S. Patent 4,198,266, issued to Kirk et al; U.S. Patent 4,431,480, issued 40 to Markham et al; U.S. Patent Number 4,220,498, issued to Prough; and an article by Kirk et al. entitled "Low-consistency Oxygen Delignification in a Pipeline Reactor - A Pilot Study", TAPPI, May 1978. Each of the foregoing describe an oxygen delignification step that operates upon pulps in the low to medium consistency range.
- U.S. Patent 4,806,203, issued to Elton, discloses an alkaline extraction, preferably for chlorinated pulp, 45 wherein the timed removal of alkaline solution is essential to prevent redepositing of dissolved lignin back onto the pulp. If too short or too long of a time period passes in this stage, the process shows little benefit. Oxygen delignification of wood pulp can be carried out on fluffed, high consistency pulp in a

weight during the oxygen delignification step. Gaseous oxygen at pressures of from about 80 to about 100 50 psig is introduced into and reacted with the high consistency pulp. See, G.A. Smook, Handbook for Pulp and Paper Technologists, Chapter 11.4 (1982). In previous oxygen delignification operations, the pulp after cooking is washed and dewatered to produce a high consistency mat. The pulp mat is then covered with a thin film or layer of an alkaline solution, by spraying the solution onto the surface of the mat. The amount of alkaline solution sprayed onto the mat is about 0.8 to 7% by weight of oven dry pulp. 55

pressurized reactor. The consistency of the pulp is typically maintained between about 20% and 30% by

Previously used high consistency oxygen delignification processes have several disadvantages. In particular, it has now been found that spraying an alkaline solution onto a mat of high consistency pulp does not provide an even distribution of solution throughout the fibrous mass, notwithstanding the generally

porous nature of such mats. As a result of this uneven distribution, certain areas of the high consistency mat, usually the outer portions, are exposed to excessive amounts of the alkaline solution. This excessive exposure is believed to cause nonselective degradation of the holocellulosic materials resulting in a relatively weak pulp, at least locally. On the other hand, other portions of the high consistency mat, typically the inner portions, may not be sufficiently exposed to the alkaline solution to achieve the desired degree of

delignification. Thus, overall quality declines.

# SUMMARY OF THE INVENTION

- The present invention provides a novel process for obtaining enhanced delignification selectivity of pulp 10 during a high consistency oxygen delignification process wherein the oxygen delignified pulp has greater strength and a lower lignin content than has been attainable by prior art processes. In addition, a wash press is utilized to reduce the quantity of alkaline material that is needed to apply the desired amount onto the pulp, while also reducing the amount of solids on the pulp which enters the oxygen delignification reactor. 15

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In accordance with the present invention, pulp is initially washed in the wash press to a consistency of at least about 18%. This consistency is subsequently reduced to less than about 10% by weight and preferably less than 5% by weight to form a low consistency pulp. A first amount of alkaline material is applied to the low consistency pulp by combining the low consistency pulp with a quantity of alkaline

- material in an aqueous alkaline solution in a manner to obtain a substantially uniform distribution of the first 20 amount of alkaline material throughout the pulp. This uniform distribution of the first amount of alkaline material is sufficient to assist in the enhancement of delignification selectivity during high consistency oxygen delignification compared to processes where the alkaline material is only applied upon high consistency pulp or is only applied at very low amounts onto low consistency pulp.
- 25 To complete the addition of the first amount of alkaline material to the pulp, the consistency of the pulp is then increased to at least about 18% to form high consistency pulp. The step of increasing the pulp consistency includes pressing or otherwise processing the low consistency pulp in a manner to remove pressate containing alkaline material while retaining the first amount of alkaline material distributed throughout the pulp.
- In one embodiment of the invention, the consistency of the pulp is increased by the wash press to a 30 value which is equal to or greater than that of the high consistency pulp. This allows all pressate to be directly recycled to the alkaline material combining step, wherein all alkaline material is applied to the low consistency pulp to distribute the total amount thereupon. Also, the quantity of alkaline material utilized to apply the total amount of alkaline material to the pulp is minimized due to the recycling of all pressate.
- In a second embodiment, a split alkaline material addition sequence is used to apply the total amount of 35 alkaline material to the pulp after it exits the wash press. A first amount of alkaline material is applied to the low consistency pulp and a second amount is applied to the high consistency pulp.

For either embodiment, a predetermined quantity of pressate may be retained in a holding tank. This guantity of pressate may be continuously returned or recycled directly to the alkaline material combining step. The total amount of alkaline material applied to the pulp is between about 0.8 and 7 percent by weight 40 based on oven dry pulp, the pulp is then subjected to oxygen delignification whereby enhanced delignification is achieved.

# BRIEF DESCRIPTION OF THE DRAWINGS

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Figure 1 is a schematic representation of one embodiment of the present invention;

Figure 2 is a schematic representation of a second embodiment of the present invention;

Figure 3 is a graph showing the relationship between pulp viscosity and K No. for softwood pulps treated with alkaline material and delignified by oxygen according to the invention compared to those representative of the prior art; and

Figure 4 is a graph showing the relationship between percent viscosity change and the proportion of alkaline material added to the high consistency pulp for pulps treated with alkaline material and delignified by oxygen according to the invention compared to pulps treated with alkaline material only at low consistency or only at high consistency.

#### DESCRIPTION OF PREFERRED EMBODIMENTS

The present invention relates to a process for treating brownstock pulp with alkaline material prior to high consistency oxygen delignification wherein the pulp is substantially uniformly treated with the alkaline material in a manner which minimizes usage of alkaline material and solids buildup on the pulp.

The present invention provides high quality, high strength, delignified wood pulp from Kraft pulp or pulps produced by other chemical pulping processes. The preferred starting material is unbleached pulp obtained by cooking wood chips or other fibrous materials in a cooking liquor, such as by the Kraft or Kraft AQ process.

- With reference to Figure 1, wood chips 1 and a white liquor 2 comprising sodium hydroxide and sodium sulfide are introduced into a digester 3. Sufficient white liquor should be introduced into the digester to substantially cover the wood chips. The contents of the digester are then heated at a temperature and for a time sufficient to allow the white liquor to substantially impregnate the wood chips and substantially complete the cooking reaction.
- This wood chip cooking step is conventionally known as Kraft cooking or the Kraft process and the pulp produced by this process is known as Kraft pulp or Kraft brownstock. Depending upon the lignocellulosic starting material, the delignification results obtained with the conventional Kraft process may be increased by the use of extended delignification techniques or the Kraft-AQ process. Moreover, these techniques are preferred for obtaining the greatest degree of reduction in K No. of the pulp without deleteriously affecting the strength and viscosity properties of the pulp during the cooking stage.
- When using the Kraft-AQ technique, the amount of anthraquinone in the cooking liquor should be an amount of at least about 0.01% by weight, based on the oven dried weight of the wood to be pulped, with amounts of from 0.02 to about 0.1% generally being preferred. The inclusion of anthraquinone in the Kraft pulping process contributes significantly to the removal of the lignin without adversely affecting the desired
- 25 strength characteristics of the remaining cellulose. Also, the additional cost for the anthraquinone is partially offset by the savings in cost of chemicals utilized in the bleaching steps which follow oxygen delignification of the pulp.

Alternatively or additively to Kraft-AQ is the use of techniques for extended delignification such as the Kamyr MCC, Beloit RDH and Sunds Super Batch Methods. These techniques also offer the ability to remove more of the lignin during cooking without adversely affecting the desired strength characteristics of the remaining cellulose.

The digester 3 produces a black liquor containing the reaction products of lignin solubilization together with brownstock 4. The cooking step is typically followed by washing to remove most of the dissolved organics and cooking chemicals for recycle and recovery, as well as a screening stage (not shown) in which the pulp is passed through a screening apparatus to remove bundles of fibers that have not been separated

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in pulping. The brownstock 4 is then directed to a blow tank 5.

Brownstock 6 exiting blow tank 5 is diluted to a consistency of about 3.5% with a portion 15A of stream 15. This pulp 6 enters wash press 7 and is washed with recycle stream 27. Instead of stream 27, other suitable wash streams which may contain alkaline materials will be apparent to persons skilled in the art. The pulp then exits the wash press 7 as stream 8 at a consistency of 25-35%. In a most preferred

embodiment, the pulp 8 has a consistency of 32%.

The use of the wash press 7 instead or in place of a conventional washer reduces the quantity of alkaline material necessary to apply the desired amount onto the pulp. Also, the amount of organic and/or inorganic solids on the pulp exiting the wash press are reduced, so that less of these contaminants are

- 45 carried by the pulp into the oxygen reactor. Thus, less amounts of chemical are consumed by the oxygen reactor. In addition, compared to a conventional washer, less alkaline material is lost to the plant liquid recovery system due to pressate discharge or due to "break through" from pressate into the washer effluent.
- The washed pulp 8 is then introduced into a mixing chest 9 where it is substantially uniformly combined with sufficient fresh 10 and recycle 14 alkaline material for a time sufficient to distribute a first amount of alkaline material throughout the pulp. During this treatment, the consistency of the brownstock pulp is reduced and maintained at less than about 10% and preferably less than about 5% by weight. The consistency of the pulp is generally greater than about 0.5%, since lesser consistencies are not economical to process in this manner. A most preferred consistency range is 0.5 to 4.5%.
- <sup>55</sup> One skilled in the art can select the appropriate quantities (i.e., concentrations and flow rates) of alkaline solution and pulp treatment times in this step to achieve a distribution of the desired amount of alkaline material throughout the pulp. In particular, an aqueous sodium hydroxide solution is combined with the low consistency pulp in an amount sufficient to provide at least about 0.8% to about 7% by weight of sodium

hydroxide on pulp based on oven dry pulp after thickening. As noted above, other alkali sources having equivalent sodium hydroxide content can also be employed, such as oxidized white liquor.

The alkaline material treated pulp 11 is forwarded to a thickening unit 12 where the consistency of the pulp is increased, for example, by pressing to at least about 18% by weight and preferably from about 25%

to 35%. For the preferred embodiment described above, the consistency is increased to 29%. The pulp consistency increasing step also removes residual liquid or pressate 13. When the consistency of the pulp 8 entering the mixing chest 9 is on the same order (i.e., about equal or slightly greater) as that of the high consistency pulp 17 which exits the thickener 12, the quantity of alkaline material utilized in the combining step is minimized because all pressate 13 may advantageously be directly recycled back to the mixing chest 9, as shown in Figure 1. The use of wash press 7 allows pulp 8 to obtain a sufficiently high

consistency so that all pressate may be recycled to the mixing chest 9 via stream 14. Alternatively, a holding tank 16 may be included to receive pressate 13. This holding tank 16 assists in the smooth, continuous operation of the process by being able to retain amounts of pressate 13 so as to provide an uninterrupted flow of pressate 14 containing alkaline material to the mixing chest 9. Thus,

<sup>15</sup> holding tank 16 provides a reservoir of alkaline material which can be continuously directed to mixing chest 9 for use in the low consistency pulp alkaline treatment step. For example, this tank should be sized at about 6000 cubic feet in order to have sufficient volume to handle the pressate generated by a 1000 air dried tons per day ("ADT/d") plant.

The addition of pressate 14 to mixing chest 9 or holding vessel 16 allows all such alkaline material to be retained within the low consistency pulp alkaline treatment stage. This avoids loss of alkaline material to the recovery system which would occur if the pressate 14 were introduced into a conventional washer by a shower or split shower configuration due to "breakthrough" into the effluent of the washer. A closed system is achieved, whereby pressate 13 is directly recycled to mixing chest 9. The amount of alkaline material "lost" due to application to the increased consistency pulp is easily replaced by additional alkaline material

10 added to the mixing chest 9 or holding tank 16. In this arrangement, the quantities of alkaline material to be utilized in the process would be minimized, since no alkaline material is lost by intentional or unintentional discharge to the plant recovery system.

A first portion 27 of the oxygen stage washer 23 filtrate 26 can be used to advantage by being recycled to wash press 7 to recover the alkaline material utilized by the process. Portions 15A, 15B of wash filtrate 30 15 from wash press 7 can be used to dilute pulp 6 or to wash pulp upstream of wash press 7. The remaining portion 15C of wash filtrate 15, if any, can be discharged to the plant recovery system to maintain water balance. The second portion 28 of oxygen stage washer 23 filtrate 26 may also be used in an analogous manner to that of filtrate 15.

- One skilled in the art would clearly recognize and understand the difference between the "quantity" of alkaline material utilized in or combined with the low consistency pulp and the "amount" which is applied to or is retained upon the pulp. To retain the desired amount of alkaline material upon the pulp after pressing, a significantly larger quantity of alkaline material must be combined with the low consistency pulp in mixing chest 9. Thus, the quantity of alkaline material which is utilized (i.e., present) in the mixing chest is always greater than the amount actually applied upon (i.e., retained within or upon) the pulp after pressing to high
- 40 consistency. Also, all alkaline material is added to the pulp in mixing chest 9 to obtain a uniform dispersal of the desired amount of alkaline material in and throughout the low consistency pulp which, after thickening, achieves the amounts applied to the pulp which are desired for high consistency oxygen delignification of the pulp. Thus, for this embodiment, alkaline material added to chest 9 at stream 10 is the same as the amount on the pulp 17 exiting thickening unit 12.
- In a second embodiment of the invention which is also shown in Figure 1, a first portion of the total amount of alkaline material desired on the pulp for the high consistency delignification in the oxygen reactor 20 is added to the low consistency pulp in the mixing chest 9. Generally, about half of the total amount is applied during the low consistency treatment described above. Thereafter, a second portion of additional alkaline material 18 is applied to the high consistency brownstock 17 produced by the thickening unit 12 by
- 50 conventional spray techniques, for example, to obtain the total amount of alkaline material on the pulp prior to oxygen delignification. This second amount of alkaline material 18 is selected to apply the remaining amount necessary (again, about one-half of the total amount) to achieve the desired extent of delignification in the subsequent oxygen delignification step which is carried out on the alkaline material treated high consistency pulp. This two-step process is referred to as the split alkaline material addition process.
- As noted above, the total amount of alkaline material actually applied onto the pulp will generally be between 0.8 and 7% by weight based on oven dry ("OD") pulp, and preferably between about 1.5 and 4% for southern softwood and between about 1 and 3.8% for hardwood. In the alternate embodiment of Figure 1, about half these amounts are preferably applied in each of the low consistency and high consistency

treatments. Thus, about 0.5 to 2% by weight, preferably about 0.5 to 1.9% for hardwood and 0.75 to 2% for softwood, is applied onto the pulp during each of the low and high consistency pulp alkaline treatments.

The split addition process improves control of the addition of alkaline material to the pulp. The high consistency alkaline treatment step allows rapid modification or adjustment of the total amount of the

alkaline material present in or upon the pulp which will enter the oxygen delignification reactor 20. By 5 adjusting the amount of alkaline material 18 applied onto the pulp during the high consistency treatment, prolonged equilibrium adjustments during the low consistency treatment are avoided. The increased speed in achieving equilibrium of the high consistency alkaline solution treatment allows for a more rapid response of the oxygen system to changing delignification requirements in that the precise total amount to be applied

to the pulp can be easily and rapidly varied to compensate for changes in the properties (i.e., wood type, K 10 No. and viscosity) of the incoming brownstock, or to vary the degree or extent of oxygen delignification for a particular pulp.

The fully alkaline treated pulp 19 is then forwarded to the oxygen delignification reactor 20 where it is contacted with gaseous oxygen 21 by any of a number of well known methods. Suitable conditions for oxygen delignification according to the present invention comprise introducing gaseous oxygen at about 80 15 to about 100 psig to the high consistency pulp while maintaining the temperature of the pulp between about 90 and 130°C. The average contact time between the high consistency pulp and the gaseous oxygen ranges from about 15 minutes to about 60 minutes.

After oxygen delignification in reactor 20, the delignified pulp 22 is forwarded to a washing unit 23 wherein the pulp is washed with water 24 to remove any dissolved organics and to produce high quality, 20 low color pulp 25. From here, pulp 25 can be sent to subsequent bleaching stages to produce a fully bleached product.

Another embodiment of the invention is illustrated in Figure 2, which illustrates a portion of the process of Figure 1. Where like components are utilized in this process, identical reference numbers have been utilized for convenience. Components not shown in Figure 2 would be identical to those of Figure 1.

In this embodiment, the alkaline material treated pulp 11 is forwarded to a wash press 30, instead of thickening unit 12, for increasing the consistency of the pulp by removing pressate 13. Also, a holding tank 16 may be provided in the recycle line for the reasons mentioned above.

There are two variations for the addition of alkaline material to the pulp by the embodiment of Figure 2. A first variation allows all alkaline material to be combined with the pulp in mixing chest 9 as described 30 above with regard to the process of Figure 1. A second variation of the Figure 2 embodiment is the use of the split alkaline material addition process of Figure 1. Here, only a portion of the desired total amount of alkaline material is applied to the low consistency pulp in the mixing tank 9, while the remaining portion 18 is applied to the high consistency pulp 17. The relative amounts of alkaline material in these portions and

the resultant advantages would be essentially the same as those described above with respect to the split 35 alkaline material addition embodiment of Figure 1.

Additional advantages of the present invention can be obtained during the subsequent bleaching of the oxygen delignified pulp 25. Such bleaching can be conducted with any of a wide variety of bleaching agents, including ozone, peroxide, chlorine, chlorine dioxide, hypochlorite or the like. When conventional chlorine/chlorine dioxide bleaching processes are used to increase the degree of brightness of the pulps which have been treated with alkaline material as described above, a substantially reduced amount of total

- active chlorine is used compared to the bleaching of pulps which are oxygen delignified by prior art techniques. The total amount of chlorine-containing chemicals utilized according to the present invention is reduced by about 15 to 35% by weight compared to the amount needed for the same starting pulp which is not treated with alkaline material at low pulp consistency. Similarly, when the chlorine/chlorine dioxide 45
- treated pulp is followed by an alkaline extraction stage, substantially reduced amounts of alkaline material are needed in this stage compared to a bleaching process for pulps which have not been uniformly combined with alkaline material at low consistency. The amount of alkaline material utilized in the extraction step would be reduced by about 25 to 40% by weight for pulp treated with alkaline material at low
- consistency as disclosed herein. 50

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In addition to providing cost advantages with respect to the reduced amounts of chemical necessary for such treatments, the process of the present invention also reduces the amounts of environmental pollutants caused by the use of chlorine, since reduced amounts of chlorine are used. Furthermore, due to the lower usage of chemicals in this portion of the system, the amount of contaminants in the waste water from the plant which is to be treated is correspondingly reduced with similar savings in waste water treatment

55 facilities and related costs.

# Examples

In order to illustrate the benefits and superior performance of the methods of the present invention, several tests were conducted utilizing the treatment procedure depicted in Figure 1.

As the term is used herein, delignification selectivity is a measure of cellulosic degradation relative to the extent of lignin remaining in the pulp and is an indication of the ability of the process to produce a strong pulp with low lignin content. Differences in delignification selectivity for oxygen delignification of a particular pulp can be shown, for example, by comparing the ratio of pulp viscosity to K No. or Kappa number. For this invention, the lignin content of the pulp may be measured by either K No. or Kappa number. One skilled in the art can recognize the difference between these values and can convert one number to the other, if desired.

The viscosity of a bleached pulp is representative of the degree of polymerization of the cellulose in the bleached pulp and as such is representative of the pulp. On the other hand, K No. represents the amount of lignin remaining in the pulp. Accordingly, an oxygen delignification reaction that has a high selectivity produces a bleached pulp of high strength (i.e., high viscosity) and low lignin content (i.e., low K No.).

#### Example 1 (Prior art high consistency pulp alkaline treatment)

Southern pine Kraft brownstock having a K No. of about 24 (Kappa number of 30.9) was pressed without alkaline solution treatment to a consistency of about 30-36% by weight to produce a high consistency mat of brownstock. The mat of brownstock was sprayed with a 10% sodium hydroxide solution in an amount sufficient to produce approximately 2.5 weight percent sodium hydroxide based on pulp dry weight. Dilution water was added in an amount sufficient to adjust the brownstock mat to about 27% consistency. The high consistency brownstock mat was then subjected to oxygen delignification using the following conditions: 110° C, 30 minutes, 80 psig O<sub>2</sub>. The oxygen delignified pulp produced according to this procedure was tested and found to have a K No. of 13 (Kappa number of 15.2) and a CED viscosity of

this procedure was tested and found to have a K No. of 13 (Kappa number of 15.2) and a CED viscosity of about 14.8 cps. This oxygen delignified pulp was further bleached by known technology. The strength and physical properties of both the oxygen delignified pulp and the fully bleached pulp are shown in Tables 1 and 2, respectively.

#### TABLE 1

	Comparison of Oxygen Stage Delignification Results on Pulps Produced by Example 1 and Example 2			
35		EXAMPLE 1	EXAMPLE 2	
	K No.	13	9	
	Viscosity (cps)	14.8	14.0	
	Ratio of Viscosity\K No.	1.14	1.55	

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5	Of P	ulps Produce	ed by Example	1 and Exam	ple 2
		EXAM	PLE 1	EXAM	IPLE 2
10	Final G.E. brightness,	* 8	3	8	33
	C.S. Free- ness, ml.	Breaking <u>Length-km</u>	Tear <u>Factor, Dm<sup>2</sup></u>	Breaking <u>Length-km</u>	Tear <u>Factor, Dm<sup>2</sup></u>
	658	6.42	55.7	7.00	55.5
75	516	8.25	73.6	8.35	67.4
	337	8.80	74.1	9.07	71.8

# TABLE 2 Comparison of Fully Bleached Strength Properties

Bleaching of the oxygen delignified pulp was conducted in three stages: chlorine, caustic extraction and chlorine dioxide. The final bleached pulp of 83 G.E. brightness was obtained using the bleaching and extraction conditions of Table 3 and the chemical charges (percent based on OD pulp) listed in Table 4. Also, the pulps were well washed between bleaching stages.

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# TABLE 3

	Bleaching Conditions in the <u>Chlorine Dioxide Stag</u> es for	Chlorine, Extract Example 1 and Exa	ion and
5	Chlorine Stage	•	
	Time, min.	15	
	Temperature, ° C	50	
10	Consistency, %	3	
	Extraction Stage		
	Time, min.	60	
15	Temperature, ° C	70	
	Consistency, %	12	
	<u>Chlorine Dioxide Stage</u>		
	Time, min.	120	
20	Temperature, ° C	60	
	Consistency, %	12	
25	TABLE	<u>4</u>	
	Bleach Chemical Usac Extraction and Chlorin	e in Chlorine, <u>e Dioxide Stages</u>	
30		EXAMPLE 1	EXAMPLE 2
	<u>Chlorine Stage</u>		
	Chlorine, % on fiber	3.6	2.4
35	Chlorine Dioxide, %	0.6	0.4
	Extraction Stage		
	Sodium Hydroxide, %	1.5	1.5
	Chlorine Dioxide Stage		
40	Chlorine Dioxide, %	0.28	0.23

Examples 2-5 (Low consistency pulp alkaline treatment)

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Examples 2-5 illustrate the benefits in degree of delignification and delignification selectivities obtained during high consistency oxygen delignification for pulps which are treated with alkaline material only at low consistency.

50 Example 2

The same pine Kraft brownstock as used in Example 1 was introduced into a mixing chest, such as 9 of Figures 1 or 2. Sufficient dilution water was added to obtain a brownstock consistency of about 3% by weight in the mixing chest. A sufficient volume of 10% NaOH solution was added to effect a 30% NaOH addition based on OD pulp. The brownstock and the aqueous sodium hydroxide solution were uniformly mixed at room temperature for about 15 minutes to combine the alkaline material with the brownstock. The resulting alkaline material containing brownstock was then pressed to a consistency of about 27% by weight. After pressing, the sodium hydroxide on the fiber equaled about 2.5%, as in Example 1. The

alkaline material treated brownstock was then bleached according to the oxygen delignification procedure described in Example 1. The oxygen delignified pulp was then washed to remove organics. The resulting oxygen stage pulp had a K No. of 9 (Kappa number of 10.8) and a CED viscosity of 14.0. The oxygen bleached pulp was further bleached by known technology at the conditions shown in Example 1. The properties of the oxygen delignified pulp and the fully bleached pulp of this Example are also shown above

5 properties of the oxygen delign in Tables 1 and 2, respectively.

As can be seen from a comparison of Examples 1 and 2, the procedure of Example 2 produced an oxygen delignified pulp having greater delignification (lower K No.) at about the same viscosity than the prior art method of Example 1 which applies all the alkaline material upon the high consistency pulp.

10 Furthermore, utilizing a low consistency alkaline treatment of pulp in accordance with Example 2 provides enhanced delignification without significant change in strength properties. Thus, increased delignification selectivity is achieved.

As a result of the lower K Nos. of pulp produced by Example 2, subsequent bleaching steps can be adjusted to accommodate the higher delignified pulp. Thus, the bleaching stages for such pulp require less bleaching agents (as shown in Table 4) or shorter bleaching times than for pulp which is not treated with alkaline material at low consistency.

# Example 3

Pulp produced from softwood (pine) in a process similar to that of Example 2 is compared to that produced conventionally (i.e. with no low consistency alkaline treatment step) as in Example 1. The average sodium hydroxide dosage applied only to high consistency pulp for subsequent oxygen delignification of the pulp was found to be 45 pounds per oven dried ton (1b/t) or 2.3%. At that level, the average reduction in K No. across the oxygen delignification reactor was 10 units. For the same level of sodium hydroxide applied only to high consistency oxygen delignification, an average K No. drop

during delignification was found to be 13 units: a 30% increase compared to the prior art.
 The average K No. and viscosity for conventional pulp was 12.1 and 14.4 cps, respectively. For the low consistency alkaline material treatment process, the average K No. at essentially the same viscosity (14.0 cps) was 8.3, an increase in delignification selectivity of about 41% (1.69 vs. 1.19), as shown in Table 5.

<sup>30</sup> Bleach plant response for pulps prepared according to the above low consistency alkaline treatment process was compared to that for pulps prepared conventionally and is shown below in Table 5.

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	Pulp Propert	y and Bleach Ch (Pine)	emical Comparison				
5	Digester	<u>Conventional</u>	Low Consistency Treated				
	DIGESCEL						
10	K No.	21.9	20.5				
	Viscosity (cps)	21.5	20.5				
15	Ratio of Viscosity/K No.	.98	1.0				
	O <sub>2</sub> Delignification S	stage					
	K No.	12.1	8.3				
20	Viscosity (cps)	14.4	14.0				
	Ratio of Viscosity/K No.	1.19	1.69				
25	Caustic, lb/t	39.4	46.0				
	Delignification (%)	44.7	59.5				
30	<u>Bleach Plant</u>						
	<u>Chlorine/Chlorine Di</u>	<u>Chlorine/Chlorine Dioxide Stage</u>					
	$Cl_2$ , $lb/t$	51.2	34.4				
35	C10 <sub>2</sub> , lb/t	7.0	4.6				
	Tot. Act. C1, lb/t	69.4	46.4				
40	Extraction Stage						
	NaOH, lb./t	35.2	23.8				
	<u>Chlorine Dioxide Ble</u>	ach Stage					
45	C10 <sub>2</sub> , lb/t	10.6	9.0				
	Viscosity (cps)	12.6	11.9				
	Dirt	5.6	2.5				

# TABLE 5

Table 5 illustrates that total active chlorine usage in the next stage of bleaching was reduced by about 1/3 (i.e., 69.4 pounds per ton vs. 46.4 pounds per ton). In addition, sodium hydroxide requirements for the extraction stage were also reduced by about 1/3 (24 lb/t vs. 35 lb/t). Chlorine dioxide in the final bleaching stage was reduced by about 1/6 (9 lb/t vs. 10.6 lb/t).

<sup>50</sup> 

# Example 4

Comparison tests similar to Example 3 were carried out for hardwood pulp. Again, it was found that a significantly larger K No. drop during the oxygen delignification reaction is achieved using a treatment <sup>5</sup> process where alkaline material is applied only to low consistency pulp compared to conventional processing. The sodium hydroxide dosage for oxygen delignification of hardwood is 27 lb/t, or 1.4%. A K No. drop of about 5 units during the delignification step was obtained for the conventional process. For the same level of sodium hydroxide utilized according to the above low consistency process, an average K No. drop of about 7.3 units was obtained, an increase of almost 50%.

- The average hardwood K No. and viscosity were found to be 7.6 and 16 cps, respectively. For the above low consistency treatment, a K No. of 6 and a viscosity of 17.7 was obtained. Also, the K No. at the same viscosity as the prior art alkaline material treated pulp (16 cps), was found to be 5.8. An increase of delignification selectivity of about 40% (2.95 vs. 2.10) is achieved, as shown in Table 6.
- Delignification selectivity can also be expressed in terms of the change in viscosity versus the change in K No. between brownstock and delignified pulps. In comparing pulps which are treated with alkaline material only at low consistency to those of the prior art, there is a greater increase in delignification selectivity for increased degrees of delignification. For a change in K No. of 4 units, the average change in viscosity was 4 cps for pulps produced by the conventional process. By contrast, the change in K No. for the same change in viscosity for pulps produced by the low consistency pulp treatment was 7 units. 20 Expressed in terms of a selectivity ratio, the selectivity for the low consistency treated pulp was 1.75 and

that for the conventional process was 1 (cps/K No.), an increase of about 75%.

A comparison of bleach plant response of oxygen delignified pulps prepared using the above low consistency alkaline material treatment in terms of bleach chemical application is compared to conventionally prepared oxygen delignified pulps in Table 6.



	Pulp Property	and Bleach Che (Hardwood)	emical Comparison
5		Conventional	Low Consistency <u>Treated</u>
	Digester		
10	K No.	12.3	13.0
	Viscosity (cps)	21.6	23.4
15	Ratio of Viscosity/K No.	1.75	1.80
	O <sub>2</sub> Delignification St	age	
	K NO.	7.6	6.0
20	Viscosity (cps)	16.0	17.7
	Ratio of Viscosity/K No.	2.10	2.95
25	Caustic, lb/t	27.6	26.4
	Delignification (%)	38.0	54.0
30	<u>Bleach Plant</u>		
	<u>Chlorine/Chlorine Di</u>	<u>oxide Stage</u>	
	$Cl_2$ , $lb/t$	27.0	22.7
35	C10 <sub>2</sub> , lb/t	5.6	4.7
	Tot. Act. C1, lb/t	41.6	34.9
40	Extraction Stage		
	NaOH, lb./t	18.9	13.3
	<u>Chlorine Dioxide Ble</u>	<u>ach Stage</u>	
45	C10 <sub>2</sub> , lb/t	5.5	4.7
	Viscosity (cps)	14.6	14.9
	Dirt	32.0	34.0

# TABLE 6

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Table 6 illustrates that total active chlorine usage in the chlorine stage was reduced by about 1/6 (i.e., 34.9 lb/t compared to 41.6 1b/t), while caustic requirements for the extraction stage were reduced by more than 29% (i.e., 13.3 lb/t vs. 18.9 lb/t) compared to prior art pulp. The chlorine dioxide in the final bleaching stage was reduced by more than 14% (i.e., 4.7 lb/t vs. 5.5 lb/t). The final pulp properties with regard to viscosity and dirt values were essentially the same.

<sup>55</sup> 

## Example 5

The following laboratory tests are included to further illustrate how to achieve a uniform distribution of alkaline material upon the pulp in accordance with the process of the present invention.

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An unbleached brownstock pine pulp was prepared having a K No. of 19.54 and a viscosity of 24.9. Two samples of this pulp at a consistency of 7.7% were treated with 3% NaOH at a temperature of 60°C for 1 and 15 minutes, respectively. Thereafter, the consistency of the pulp was increased to 27% and the NaOH content of the pulp was found to be about 0.67%. This pulp was directed to an oxygen delignification reactor at a pressure of 80 psi and a temperature of 110°C for 30 minutes without the further addition of alkaline material.

Next, two additional samples of the unbleached pulp, each at a consistency of 3%, were treated with a NaOH application of about 35% at a temperature of 60°C for 1 and 15 minutes, respectively. Thereafter, the consistency of the pulp was increased to 27%, while retaining a NaOH content of 3% throughout the pulp, and the pulp was directed to an oxygen delignification at a pressure of 80 psi and a temperature of 10°C for 1 and 15 minutes.

15 110°C for 30 minutes without the further addition of alkaline material. The results are shown in Table 7 below:

TABLE	7
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20	Sample	Consistency %	Mixing Time (minutes)	Properties after Oxygen Delignification	
				K No. (25 ml)	Viscosity (cps)
	A	7.7	1	17.37	23.2
25	В	7.7	1	17.43	22.6
	С	7.7	15	17.77	24.3
	D	7.7	15	17.34	22.0
22	E	3.0	1	8.74	14.8
	F	3.0	1	8.34	14.8
30	G	3.0	15	8.24	15.3
	Н	3.0	15	8.73	14.3

The treated pulp of samples E-H retains a much greater amount (i.e., 3%) of sodium hydroxide than that of samples A-D, because a much larger quantity of sodium hydroxide is mixed with the pulp. Samples E-H show a decrease in K No. of the pulp of at least about 55.3%, while the K No. decrease of Samples A-D is much smaller and is, at best, about 11.3%. Thus, the samples (E-H) treated in accordance with the process of the present invention increases delignification by about 49.6% over the comparative samples.

For the same unbleached brownstock pulp of this example, the preceding tests were repeated with the following changes:

	Modification 1	Modification 2
1st Stage: NaOH, % on pulp	3	24
Consistency, %	3.5	3
Temperature, °C	48	48
Oxygen stage: NaOH, % on pulp	0.44	3
Consistency, %	20	20

<sup>50</sup> The NaOH treatment time for each modification was conducted both at 2 minutes and 15 minutes. As noted, the consistencies of the unbleached pulp were essentially the same (3.5% vs. 3%). Results are shown in Table 8.

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5	Sample	Consistency %	Mixing Time (minutes)	Properties after Oxy	Properties after Oxygen Delignification	
				K No. (25 ml)	Viscosity (cps)	
	I	3.5	2	15.75	23.4	24.8
	J	3.5	2	15.34	22.4	25.2
40	К	3.5	15	14.78	22.6	25.9
10	L	3.5	15	15.00	22.7	25.5
	М	3.0	2	8.59	13.3	36.6
	Ν	3.0	2	8.29	14.2	35.3
	0	3.0	15	8.14	13.1	36.3
15	Р	3.0	15	8.44	13.8	36.5

# TABLE 8

Due to the increased amount of NaOH mixed with the low consistency pulp, a much greater amount of NaOH is retained on the high consistency pulp. Due to this increased amount of NaOH, samples M-P achieve a decrease in K No. of at least about 56%, while samples I-L, at best, achieve a decrease of only about 24.4%. Again, the samples (M-P) prepared by the present process obtain increased delignification by 20 at least 41.9% compared to the comparative samples. As noted above, this is due to the increased amounts of sodium hydroxide retained upon the high consistency pulp due to the uniform mixing and distribution of appropriate amounts of sodium hydroxide throughout the low consistency pulp.

#### 25 Example 6

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To illustrate the effect of 100% low consistency alkaline material treatment on pulp prior to oxygen delignification and its contribution to the overall effectiveness of Kappa drop and total yield, the Kappa number and yield were determined for both conventional and low Kappa number Kraft/AQ brownstocks. The results are presented in Table 9.

35	Brownstock	Time (Min.)	LOW CONSISTENCY ALKALINE TREATMENT		OXYGEN DELIGNIFICATION			
			Initial Kappa Number	Final Kappa Number	Yield (%)	Kappa Number	Yield (%)	Final Viscosity (CPS)
40	<sup>1</sup> Conven. <sup>2</sup> Conven. <sup>3</sup> K/AQ <sup>4</sup> K/AQ	5 15 5 5	28.1 28.1 21.6 21.6	26.5 27.5 20.3 	99.5 98.7 100.0 	12.0 13.4 8.9 8.1	95.2 96.3 96.7 97.2	14.7 15.1 15.2 13.9

#### TABLE 9

<sup>1</sup> 2.4% NaOH

<sup>2</sup> 2.1% NaOH 45

3 2.1% NaOH

4 2.6% NaOH

For a conventional kraft brownstock having a Kappa number of 28.1 treated with sodium hydroxide 50 (2.4% on pulp after pressing) at 3% consistency for 5 minutes, the starting Kappa number decreased 1.6 units to a post treated Kappa number of 26.5. This represented a 9.6% contribution to the total Kappa number drop experienced following alkaline treatment and oxygen delignification (Kappa number of 12.0). The yield across the low consistency alkaline treatment stage was 99.5%. Approximately half of the 0.5% loss in yield can be attributed to loss of lignin with the remainder due to a loss in carbohydrates. The total 55 yield after oxygen delignification was 95.2%.

The same starting brownstock was treated with sodium hydroxide (2.1% on pulp after pressing) at 3% consistency for 15 minutes. The starting Kappa number decreased 0.6 units to a Kappa number of 27.5. This represented a 4.2% contribution to the total Kappa number drop experienced following low consistency alkaline treatment and oxygen delignification (Kappa number of 13.4). The yield across the alkaline treatment stage was 98.7%.

For a low Kappa number kraft/AQ brownstock treated with sodium hydroxide (2.11% on pulp after pressing) at 3% consistency for 5 minutes, the Kappa number decreased 1.3 units to 20.3. This Kappa

- 5 number drop represented a 10% contribution to the total Kappa number drop experienced following oxygen delignification (Kappa number of 8.9). There was essentially no yield loss detected across the alkaline treatment stage. The total yield loss following oxygen delignification was 96.7%. A second oxygen delignification of the same kraft/AQ starting brownstock resulted in a similar Kappa number of 8.1 and yield of 97.2%.
- This Example 5 shows that no significant amount of delignification occurs during the low consistency alkaline treatment of the pulp. This example also shows that there is no significance to the time of treatment with alkaline material at low consistency up to about 15 minutes. As is further shown by Examples 2-6, however, the low consistency alkaline treatment does significantly increase the relative amount of delignification obtained during subsequent high consistency oxygen delignification step as compared to pulps
- <sup>15</sup> treated in the conventional manner. This example also shows that the process is effective with a low Kappa number brownstock in taking the pulp to a very low Kappa number without any significant decrease in viscosity.

The uniform distribution of the alkaline material throughout the pulp during the low consistency combining step ensures that the pulp fibers are more optimally associated with the alkaline material than is

- 20 otherwise possible according to prior techniques. This results in enhanced delignification selectivity during subsequent oxygen delignification in that the delignified brownstocks have strength and degrees of delignification that are generally superior to those attainable by the prior art. Also, the delignification selectivity of the oxygen delignification reaction is unexpectedly improved.
- For the present invention, the minimum amount of alkaline material applied onto the low consistency pulp is that which, in combination with the amount applied onto the high consistency pulp, is sufficient to increase or enhance delignification selectivity of the pulp during the oxygen delignification stage. As shown in the following Examples, at least about 50% of the total amount of alkaline material to be applied to the pulp prior to oxygen delignification should be applied to the low consistency pulp. If less than about 50% is applied to the low consistency pulp, the advantages regarding delignification selectivity significantly decrease.

When alkaline material is applied only to high consistency pulp as in the prior art, a delignification (i.e., reduction in K No.) of up to 50% can be achieved without substantially damaging the cellulose portions (and thus without substantially reducing the strength) of the pulp. In the present invention, it is possible to obtain a reduction in K No. for the incoming pulp of greater than 50% and generally at least about 60% during

35 oxygen delignification with essentially no damage to the cellulose portion of the pulp. Reductions of 70% and more can be achieved, if desired.

For example, upon entering the oxygen delignification stage, pulp K Nos. for the particular pulp range from about 10 to 26, depending upon the type of wood and type of pulping conducted upon the particular wood. After delignification, the K No. is reduced to about 5 to 10. For softwood pulp, K Nos. generally range

40 from 20-24 (target of 21) prior to delignification, while after delignification, the K Nos. are in the range of 8-10. For hardwood pulp, K Nos. of 10-14 (target 12.5) prior to delignification and K Nos. of 5-7 after delignification are generally obtained by the present process.

For either type of pulp, the viscosity prior to delignification is generally about 19 or greater, while after delignification is above about 13 (generally 14 or above for softwood and 15 or above for hardwood). Typically, this change in viscosity from before to after delignification would be about 6 cps. or less.

Moreover, it has been found that the change in viscosity per change in K No. is a constant for decreases in K No. up to about 17 units.

Thus, delignification selectivity is enhanced by the 100% low consistency alkali treatment process, with an increase of at least 20% in delignification compared to prior art delignification processes. The avoidance of deterioration of the cellulose component of the pulp is evident by the minimal change in viscosity of pulp from before to after oxygen delignification.

# Example 7

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55 The following data illustrates the relative quantities of alkaline material needed to treat Southern pine brownstock pulp prior to high consistency oxygen delignification.

In order to obtain about 2.2% by weight sodium hydroxide based on OD pulp at a consistency of about 27%, about 40 pounds of chemical per ton are required. For the prior art process of Example 1, this amount

is all that is needed, since it is applied directly to the high consistency pulp and 100% of the applied alkaline material enters the oxygen reactor. For the alkaline treatment of low consistency pulp as described in Example 2, it has been determined that about 35 to 54% of the quantity of the alkaline material utilized in the mixing tank is eventually applied to the pulp and enters the oxygen reactor. This indicates that as much

- as 45 to 91 pounds per ton of sodium hydroxide must be utilized to achieve the desired amount on the 5 pulp. As noted above, however, the process of Example 2 provides enhanced delignification without a significant change in strength properties, so that the additional quantities of alkaline material would be tolerated. The process illustrated in Figure 1, however, would also utilize only the necessary 40 pounds per ton, since no alkaline material is discharged from the pressate recycle and the only alkaline material which
- exits the system is that which enters the oxygen reactor. Thus, the improved delignification selectivity of the process of Example 2 is achieved with the savings of about 51 pounds per ton of alkaline material used in the process of the present invention.

#### Example 8

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The following examples of the invention illustrate how the present invention achieves delignification selectivities comparable to the 100% low consistency pulp alkaline treatment process of Examples 2-5 while reducing the amount of alkaline material removed to the recovery system by use of a split alkaline material addition process.

The following experiment involving 6 samples illustrates the effect on delignification selectivity of the 20 two step split addition process of the present invention. Results are set forth in Tables 8 and 9. For comparison purposes, samples A (100% alkali applied to low consistency pulp) and B (100% alkali applied to high consistency pulp), were included in the Tables.

The starting brownstock used in the experiment was Southern pine. This material was digested in a conventional manner to form brownstock. The 40 ml K No. of the brownstock was 22.1, and the 25 ml K No. 25 was 19.8. The viscosity of the pulp was 24.5 cps.

This pulp was diluted to a low consistency of about 3.5%. A sufficient amount of alkaline material was distributed throughout this pulp by the addition of oxidized white liquor solution. The pulp consistency was then increased to about 27% to retain, after pressing, the amount of alkaline material throughout the pulp shown in Table 10.

A second amount of alkaline material, also shown in Table 10, was then applied to the high consistency pulp. The alkali solution used to apply the stated amounts was oxidized white liquor containing 84.5 g/l sodium hydroxide and 0.1% magnesium sulfate.

The alkaline treated high consistency pulp was then directed to the oxygen reactor 20 (Figure 1) which was operated at a temperature of 110°C, at a pressure of 80 psig for 30 minutes. The total alkaline material 35 applied in both the low and high consistency pulp treatments ranged from about 2.96 to 4.23% as shown in Table 10. The actual splits of alkaline material on pulp between the low and high consistency pulp treatments are shown in Table 10, while the resulting viscosities, K Nos. and selectivity ratios for the oxygen delignified pulp are shown in Table 11.

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45	Sample	Low Consistency Alkali Addition (% on pulp)	High Consistency Alkali Addition (% on pulp)	Total Alkali Addition (% on pulp)
	A	3.10	0	3.10
	1	2.33	0.63	2.96
	2	2.25	1.17	3.42
	3	1.81	1.80	3.61
50	4	1.39	2.34	3.73
	5	1.06	2.92	3.98
	6	0.63	3.60	4.23
	В	0	4.50	4.50

# TABLE 10

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	Sample	% Added at High Consistency	Viscosity (cps)	K No. (25 ml)	Ratio of Viscosity to K No.
5	Α	0	14.9	10.1	1.475
	1	21.4	15.1	9.65	1.565
	2	34.3	13.7	9.96	1.376
	3	49.8	15.3	10.08	1.518
	4	62.7	14.0	10.66	1.313
10	5	73.4	14.3	11.82	1.210
	6	85.2	13.9	11.16	1.246
	В	100	14.4	12.80	1.125

#### TABLE 11

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The results show that the samples applying up to 49.8% (i.e., about 50%) of the alkaline material to the 15 high consistency pulp provides enhanced delignification and selectivity ratios in that lower K Nos. are achieved at equal or higher viscosities. Samples 1, 2 and 3 provide delignified pulps which are comparable to that of comparative sample A, where 100% of the alkaline material is applied to low consistency pulp. Samples 1-3 and A are preferred due to the increased delignification selectivities compared to samples 4-6 and B, viscosity decreases while K Nos. increase. Further bleaching of the pulps of samples 4-6 and B would require additional bleaching chemical compared to the pulps of samples 1-3 and A due to the higher K Nos. of the pulps of samples 4-6 and B. These results demonstrate that split alkaline additions of at least 50% in the low consistency stage retain the enhanced delignification achievable by the addition of all alkaline material to the low consistency pulp.

#### 25 Example 9

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The data presented in Examples 2 through 5 and 7, along with numerous other predicted and observed values, have been compiled for softwood pulp in graphical form in Figures 3 and 4. Figure 3 also includes curves generated from combined data from actual tests, and numerous other predicted and observed results, which illustrates the relationship of viscosity to K No. for softwood from the prior art pulp treatment process of Example 1.

As shown in Figure 3, the prior art process of Example 1 achieves typical pulp properties after oxygen delignification defined by the curve labeled Prior Art. It is desirable to maintain pulp strength, as measured by viscosity, at higher viscosity levels, while achieving effective delignification as measured by a decrease

35 in K No. Figure 3 illustrates that enhanced delignification (lower K Nos.) may be attained at a given viscosity value according to the curve representing the invention, for a low consistency pulp alkaline material treatment as compared to the lesser delignification and viscosity values according to the Prior Art curve.

- Figure 4 illustrates the effect of increasing the percentage of alkaline material utilized in treating the high consistency pulp. The solid horizontal line proximate to the 0 viscosity change numeral corresponds to 40 the baseline viscosity achieved with 100% of the alkaline material applied on the low consistency pulp. The two broken horizontal lines on either side of the solid 0 line delineate the boundaries of a typical ± 6% deviation in viscosity. As is evident from Figure 4, as the amount of alkaline material added to the high consistency pulp exceeds about 50% of the total alkaline material applied in pulp treatment, viscosity of the pulp drops below the acceptable deviation. 45
- As high consistency treatment of the pulp increases in percentage, there is consequently less alkaline material utilized in low consistency treatment. It is within the low consistency treatment step that the substantially uniform application of alkaline material onto the pulp is accomplished. As less alkaline material is available for the low consistency step, the selectivity advantages of low consistency treatment are diminished. Thus, any split addition process achieves some improvement in delignification selectivity 50
- compared to the application of all alkaline material to the high consistency pulp. The best results in delignification selectivities are achieved for a split addition where no more than about 50% of the total alkaline material is added to the high consistency pulp.

#### Example 10 55

It has been found that for Southern Pine Kraft brownstock, a target value of 2.4% based on oven dry pulp of sodium hydroxide on the pulp is needed prior to oxygen delignification to obtain the desired delignification level. In order to provide 2.4% of sodium hydroxide on the pulp entering the oxygen reactor,

approximately 43.2 pounds per air dried ton (Ib/ADT) of sodium hydroxide is required.

The amount of alkaline material lost due to the discharge of various portions of pressate is illustrated in Table 12.

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# TABLE 12

LB/ADT ALKALINE	LB/ADT ALKALINE MATERIAL APPLIED TO PULP PRIOR TO OXYGEN DELIGNIFICATION							
Pressate Discharged To Recovery (%)	Split (%) of alkaline material added to low consistency pulp							
	100% 80% 60% 50%							
0	0 43.2 43.2 43.2							
20	54      51.8      50.0      48.6        72      66.2      60.5      57.6							
40								
60	108	95.0	82.1	75.6				

It should be noted that the values listed in Table 10 refer to the total amount of alkaline material applied to pulp by the process: i.e., the amount applied by the low, consistency treatment plus the amount applied 20 to the high consistency pulp (if applicable). The 50% split column at zero pressate discharge thus indicates that 21.6 lb/ADT are applied to the low consistency pulp in the mixing chest and 21.6 lb/ADT are applied to the high consistency pulp. The same 50% split at 20% pressate discharge shows that in addition to the 21.6 lb/ADT applied to the low consistency pulp, an additional 5.4 lb/ADT must be added to the system (a total of 27 lb/ADT) to compensate for the amount lost by pressate discharge. This additional amount is generally added to the mixing chest in order to maintain the amount applied to the high consistency pulp at

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no more than about 50% of the total amount. Table 13 illustrates the same data of Table 12, but quantifies the amount of additional alkaline material that should be added to the low consistency treatment to achieve the target 2.4% NaOH on the pulp. As the percentage of alkaline material applied to the high consistency pulp increases up to 50%, less additional

30 alkaline material must be added to the low consistency treatment to maintain the proper amount of alkaline material on the pulp available for high consistency oxygen delignification. With zero pressate discharge, as described above, in particular with respect to the embodiment of Figure 1, no alkaline material is lost with the appropriate savings in chemical realized for the particular design.

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# TABLE 13

	LB/ADT ALKALINE MATERIAL APPLIED TO LOW CONSISTENCY PULP TO COMPENSATE FOR PRESSATE DISCHARGED						
40	Pressate Discharged To Recovery (%)	Split (%) of alkaline material added to low consistency pulp					
		100%	80%	60%	50%		
45	20 40 60	10.8 28.8 64.8	8.6 23 51.8	6.8 17.3 38.9	5.4 14.4 32.4		

Table 14 illustrates the same data of Tables 12 and 13, but presents only the amount of alkaline material (and corresponding weight percentage in parentheses) which is added to the low consistency pulp 50 for 20, 40 and 60% of pressate discharged.

TABL	.E 14
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lb/ADT	(% of total) Alkaline	e Material Applied	to Low Consistency	Pulp		
Pressate Discharged (%)	Split (%) of alkaline material added to low consistency pulp					
	100%	80%	60%	50%		
0	43.2 (100%)	34.6 (80%)	25.9 (60%)	21.6 (50%)		
20	54 (100%)	43.2 (83.4%)	32.7 (65.4%)	27 (55.5%)		
40	72 (100%)	57.6 (87%)	43.2 (71.4%)	36 (62.5%)		
60	108 (100%)	86.4 (90.9%)	64.8 (73.79%)	54 (71.4%)		

These data show that using the split alkaline material addition process of the invention, at least 50% and preferably about 55 to about 90% of the total amount of alkaline material is added to the low consistency pulp in mixing chest 9 to compensate for amounts of alkaline material removed to the recovery system by pressate discharge. The balance of the alkaline material is added to the high consistency pulp.

- Examining the values corresponding to 100% alkaline material applied to the low consistency pulp, it is expected, and the results indicate, that as the percentage of alkaline material lost to pressate discharge increases, a corresponding increase in alkaline material added to the pulp is necessary. For the situation where all alkaline material is combined with the low consistency pulp, the amount of alkaline material in the pressate discharge 15C sent to the recovery system is significantly higher than when only a portion of the total alkaline material is utilized during the low consistency treatment. As the percentage of alkaline material applied to the low consistency pulp decreases due to the split addition, the amount of additional alkaline material that must be added to replace alkaline material lost in the pressate discharge diminishes, because
  - less alkaline material is utilized in the low consistency treatment.

Thus, applying lesser proportions of the alkaline material onto the low consistency pulp reduces the quantity of alkaline material utilized in the mixing chest 9 and also reduces the amount of alkaline material removed via pressate discharge. This splitting of the alkaline material applied to low and high consistency pulp reduces the amount of pressate discharge 15C which in turn reduces the amount of alkaline material which must be reintroduced, thus saving chemical.

While it is apparent that the invention herein disclosed is well calculated to fulfill the objectives stated above, it will be appreciated that numerous modifications and embodiments may be devised by those skilled in the art. It is intended that the appended claims cover all such modifications and embodiments as fall within the true spirit and scope of the present invention.

#### Claims

**1.** A process for obtaining enhanced delignification selectivity of pulp during high consistency oxygen delignification which comprises:

initially washing the pulp in a first wash press to a consistency of at least about 18%;

applying a first amount of alkaline material to the washed pulp by reducing the consistency of the washed pulp to less than about 10% by weight to form low consistency pulp, combining the low consistency pulp with a quantity of alkaline material to obtain a substantially uniform distribution of alkaline material throughout the pulp, and increasing the consistency pulp and to remove pressate while retaining the first amount of alkaline material substantially uniformly distributed throughout the high consistency pulp;

- recycling a substantial portion of the pressate directly to the alkaline material combining step;
- providing a total amount of alkaline material on the high consistency pulp of at least about 0.8 to 7 percent by weight based on the oven dry weight of the pulp, said total amount including said first amount; and

oxygen delignifying the alkaline material containing high consistency pulp to obtain enhanced delignification selectivity and oxygen delignified pulp.

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2. The process of claim 1 wherein substantially all pressate is recycled to the alkaline material combining step.

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- **3.** The process of claim 1 wherein all alkaline material is applied to the reduced consistency pulp so that the total amount of alkaline material provided on the high consistency pulp is equal to the first amount.
- 4. The process of claim 1 which further comprises applying a second amount of alkaline material onto the high consistency pulp wherein the combination of the first and second amounts equals the total amount of alkaline material provided on the high consistency pulp.
  - 5. The process of claim 1 wherein the consistency of the pulp is increased a second wash press.

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- **6.** The process of claim 1 which further comprises holding a predetermined quantity of pressate in a holding tank in order to continuously return pressate directly to the alkaline material combining step in the event of intermittent or non-continuous operation of the consistency increasing step.
- The process of claim 1 which further comprises washing oxygen delignified pulp, thus generating wash
  water effluent, and recycling a portion of the wash water effluent to the initial pulp washing step.
  - 8. The process of claim 1 wherein the pulp has a low consistency of less than about 5% by weight when combined with the quantity of alkaline material.
- **9.** The process of claim 1 wherein the consistency of the pulp is increased to between about 25 and 35% by weight prior oxygen delignification.
  - **10.** The process of claim 1 wherein the oxygen delignifying step obtains enhanced delignification selectivity by decreasing the K No. of the high consistency pulp by greater than 50% without significantly damaging the cellulose components of the pulp.
  - **11.** The process of claim 10 wherein the K No. is decreased from about 10 to 26 before delignification to about 5 to 10 after delignification.
- **12.** The process of claim 4 wherein the pulp is unbleached softwood pulp and the total amount of alkaline material applied to the pulp is between about 1.5 and 4 percent by weight, with the first and second amounts each ranging between about 0.75 and 2 percent by weight.
- 13. The process of claim 4 wherein the pulp is unbleached hardwood pulp and the total amount of alkaline material applied to the pulp is between about 1 and 3.8 percent by weight, with the first and second amounts each ranging between about 0.5 and 1.9 percent by weight.
  - **14.** A process for obtaining enhanced delignification selectivity of pulp during high consistency oxygen delignification which comprises:
  - initially washing the pulp in a first wash press to a consistency of at least about 18%;

applying a first amount of alkaline material to the washed pulp by reducing the consistency of the washed pulp to less than about 10% by weight to form low consistency pulp, combining the low consistency pulp with a quantity of alkaline material to obtain a substantially uniform distribution of alkaline material throughout the pulp, and increasing the consistency of the pulp in a second wash press to at least about 18% by weight to obtain high consistency pulp and to remove pressate while retaining the first amount of alkaline material substantially uniformly distributed throughout the high consistency pulp;

recycling a substantial portion of the pressate to the alkaline material combining step;

providing a total amount of alkaline material on the high consistency pulp of at least about 0.8 to 7 percent by weight based on the oven dry weight of the pulp, said total amount including said first amount; and

oxygen delignifying the alkaline material containing high consistency pulp to obtain enhanced delignification selectivity and oxygen delignified pulp.

**15.** The process of claim 14 wherein all alkaline material is applied to the reduced consistency pulp so that the total amount of alkaline material provided on the high consistency pulp is equal to the first amount.

- **16.** The process of claim 14 which further comprises applying a second amount of alkaline material onto the high consistency pulp wherein the combination of the first and second amounts equals the total amount of alkaline material provided on the high consistency pulp.
- 5 **17.** The process of claim 14 which further comprises holding a predetermined quantity of pressate in a holding tank in order to continuously return pressate directly to the alkaline material combining step in the event of intermittent or non-continuous operation of the consistency increasing step.
- 18. The process of claim 14 which further comprises washing the oxygen delignified pulp, thus generatingwash water effluent and recycling a portion of the wash water effluent to the first wash press.
  - **19.** The process of claim 16 wherein the pulp is unbleached softwood pulp and the total amount of alkaline material applied to the pulp is between about 1.5 and 4 percent by weight, with the first and second amounts each ranging between about 0.75 and 2 percent by weight.
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- **20.** The process of claim 16 wherein the pulp is unbleached hardwood pulp and the total amount of alkaline material applied to the pulp is between about 1 and 3.8 percent by weight, with the first and second amounts each ranging between about 0.5 and 1.9 percent by weight.
- 20 21. The process of claim 14 wherein the oxygen delignifying step obtains enhanced delignification selectivity by decreasing the K No. of the high consistency pulp by greater than 50% without significantly damaging the cellulose components of the pulp.
  - **22.** The process of claim 21 wherein the K No. is decreased from about 10 to 26 before delignification to about 5 to 10 after delignification.

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K NUMBER (40 ml)

FIG. 3





FIG. 4



European Patent Office

# EUROPEAN SEARCH REPORT

Application Number

EP 92 20 2469

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Category	Citation of document with ind of relevant pass	cation, where appropriate, ages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
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	Place of search	Date of completion of the	search	Examiner
ſ	HE HAGUE	24 NOVEMBER 1	992	BERNARDO NORIEGA
( X : part	CATEGORY OF CITED DOCUMENT	S T : theory E : earlier after ti	or principle underlying the patent document, but publ he filing date	invention ished on, or
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