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(54) **Title**: METHOD OF BLEACHING A PULP

(57) **Abstract**: A bleaching method for bleaching a hardwood pulp to a brightness of 88- 92 % ISO is disclosed. The method comprises subjecting an oxygen delignified pulp to a hot chlorine dioxide bleaching step followed by an integrated alkaline extraction and chloride dioxide bleaching step.

METHOD OF BLEACHING A PULP

The present invention relates to a method of bleaching of a pulp. More specifically, it relates to a method of bleaching an oxygen delignified pulp, such as an oxygen delignified hardwood pulp, to a brightness of 88-92 % ISO.

Background

In bleaching processes for both softwood and hardwood pulps, the pulps are normally delignified in one or more oxygen steps and thereafter bleached by means of various sequences comprising chlorine dioxide steps, extraction steps, peroxide steps etc.

Hardwood pulps differ from softwood pulps in that they contain high amounts of Hexenuronic Acid (HexA). The amount of HexA depends on the raw material used and the cooking conditions. Modern methods of cooking, which utilize relatively low cooking temperatures, normally render high contents of HexA. HexA is oxidized by potassium permanganate ($KMNO_4$) and thereby contributes to the kappa number. In a hardwood pulp with a kappa value of 10, 50-70% of the kappa value could be a result of HexA and only 30-50% is attributed to lignin and other compounds.

During bleaching, HexA can be reduced by oxidation with bleaching chemicals such as chlorine dioxide and ozone. A more economical way is to degrade HexA by means of acid hydrolysis at high temperature, which lowers the amount of double bonds in the remaining pulp. Therefore, a hot chlorine dioxide step (D_{HT}) is often accommodated in modern bleach plants. In this stage both oxidation and acid hydrolysis are performed. The high temperature in D_{HT} can give a reduction of the kappa number from for example 10.5 to 2.5. Hence, most of the reduction of the kappa number, typically 85-90%, is achieved in such a D_{HT}-step and only a minor part, typically 10-15 %, in a following extraction step (E). Moreover, it is believed that lignin is also degraded into smaller, more water soluble pieces during the D_{HT}-step.

SE 466 062 discloses a method of bleaching chemical pulp in a sequence comprising at least four bleaching steps with final bleaching in a first and a second chlorine dioxide step. Between the chlorine dioxide steps an alkaline extraction is carried out and washing takes place between the first chlorine dioxide step and

extraction. Immediately after said washing step, NaOH is charged in an amount of 4-10 kg/ton pulp. Thereafter an oxidizing agent is admixed in an amount of up to 2 kg/ton pulp. An acid is added for lowering the pH-value, but without effecting a complete neutralization of residual alkaline.

SE 526 162 discloses a bleaching process for hardwood pulp wherein an oxygen-delignified and washed pulp is subjected to a chlorine dioxide bleaching step at high temperature, such as at least 90 °C, and treated with a chelating agent without any intermediate wash. The pulp is thereafter washed and subjected to a pressurized peroxide bleaching step in which alkali is also added. The bleached pulp is finally washed in order to obtain a pulp with a brightness of 88-90 % ISO.

WO 02/075046 discloses a method for end bleaching of pulp comprising two chlorine dioxide steps. The pulp is washed and dewatered after the first chlorine dioxide step to a concentration of 12-50 % in order to remove dissolved metal ions. Thereafter, alkali is added for extraction and rapid increase of the pH. Before the pulp is introduced into the second chlorine dioxide step, acid and chlorine are added to the pulp.

The previously known methods generally perform well, even though they may be fairly expensive or complex. Nonetheless, there is always a desire to further improve the bleaching, especially for hardwood pulps, and reduce the overall costs for the bleaching.

Hence, the object of the invention is to provide a method for bleaching a hardwood pulp to a brightness of 88-92 % ISO in a cost effective manner.

Summary

The above identified object is achieved by means of the method of bleaching a pulp in accordance with independent claim 1. Preferred embodiments are given in the dependent claims.

The method of bleaching a pulp comprises subjecting an oxygen delignified pulp to a hot chloride dioxide bleaching step at a temperature of 80-95 °C and a pH of 2-4 followed by washing. During said bleaching step, a substantial reduction of the kappa number will be accomplished. The pulp is thereafter subjected to an alkaline extraction step and a chlorine dioxide bleaching step integrated with said alkaline extraction step. In the present disclosure, integrated

should be interpreted as following directly after the preceding step without any intermediate wash.

It has been determined that it is possible to obtain a brightness of more than 88 % ISO when bleaching a hardwood pulp by means of the method according to the invention. Furthermore, excellent reverted brightness can be achieved. The COD generation is also reduced compared to bleaching methods according to previous known methods used to obtain the same brightness. Moreover, the overall cost for bleaching a hardwood pulp is reduced as a consequence of lower chemical costs and/or lower investment costs for the bleaching plant, mainly as a result of fewer required washing steps.

Even though the method according to the invention is intended for bleaching hardwood pulp, it is also believed to be suitable for bleaching softwood pulp.

Detailed description

In accordance with the present invention, an oxygen-delignified and washed pulp is subjected to a hot chlorine dioxide step (D_{HT}) in a reactor in order to reduce the kappa value to typically 3 or less. The hot chlorine dioxide step is performed at a temperature of 80-95 °C, preferably 85-95 °C, on a pulp having a consistency of 8-20%, preferably 8-15 %, at a pH of 2-4, preferably pH 2.5-3.5, for a period of time sufficient to reduce the kappa number to the desired value. It should be noted that the time required for achieving the desired result depends on selected values of the parameters given above. However, the skilled person can easily determine the suitable period of time for the selected parameters by routine tests.

After the hot chlorine dioxide step the pulp is washed in accordance with conventional techniques, for example by using a wash-press or a dewatering-press, in order to remove the dissolved matter.

Alkali, for example in the form of a liquid containing NaOH, is thereafter added to the pulp in order to subject the pulp to an alkaline extraction step at a pH of 8-14, preferably pH 9-12, for a period of time sufficient to dissolve oxidized lignin. The consistency of the pulp should in this step be 8-20 %, preferably 8-15%. The alkaline extraction step may suitably be performed at a temperature of 75-85 °C for 2-30 minutes, preferably 5-15 minutes.

Chlorine dioxide is added to the pulp directly after said alkaline extraction step, i.e. without any intermediate wash, and the pH of the pulp is adjusted to 2-4, preferably pH 2.5-4. This chlorine dioxide addition will subject the pulp to a second chlorine dioxide bleaching step. The temperature of the pulp should preferably be the same, or substantially the same, in this second bleaching step as in the alkaline extraction step. Since there is no washing step between the alkaline extraction step and the second chlorine dioxide bleaching step, these are considered to be integrated steps.

After the second bleaching step, the pulp may be subjected to a peroxide treatment. This may be performed directly after the second bleaching step, i.e. integrated with the alkaline extraction and chlorine dioxide bleaching step, or after an intermediate washing step. The peroxide treatment is performed at a temperature of 75-90 °C for a period of time sufficient to accomplish the desired final brightness, such as 88-92 % ISO, after subsequent wash of the pulp. It should be noted that the time required for achieving the desired result depends on the amount peroxide used and the temperature of the pulp given above, but can easily be determined by the skilled person by routine tests.

According to an alternative embodiment of the bleaching method of the invention, the alkaline extraction step and the second chlorine dioxide bleaching step are repeated after an intermediate wash.

The amount of chemicals required in each step of the process according to the invention to obtain the desired result can be easily determined by the skilled person by using common general knowledge within the field of bleaching or by mere routine tests.

It has been noted that by using a sequence comprising a hot chloride dioxide bleaching step followed by an integrated alkaline extraction and chlorine dioxide bleaching step in accordance with the present invention, it is possible to obtain a brightness of 89 % ISO when bleaching a hardwood pulp. By repeating said integrated alkaline extraction and chlorine dioxide bleaching step in such a sequence, it is possible to obtain a brightness of about 92 % ISO. Moreover, 92 % ISO can also be obtained by using a sequence comprising a hot chloride dioxide bleaching step followed by an integrated alkaline extraction and chlorine dioxide bleaching step and a subsequent peroxide step in accordance with a preferred embodiment of the invention.

The bleaching method according to the invention has proven to be especially suitable for bleaching Eucalyptus-based pulps.

Example 1

A sulphate pulp produced from *Eucalyptus grandis* wood was used for laboratory tests. The unbleached pulp had a kappa number of 18. After oxygen delignification, the pulp had a kappa number of 10.5, a viscosity of 1090 ml/g and a brightness of 65 % ISO.

The pulp was bleached with two different sequences according to the invention, S_{inv1} and S_{inv2}, and two reference sequences, S_{Ref1} and S_{Ref2}. The sequences (S_{inv1}, S_{inv2}, S_{Ref1}, S_{Ref2}) are listed below and the results are shown in Table 1.

S_{Ref1}

- Chlorine dioxide bleaching of a pulp with 10 % consistency at 90 °C and pH 2.6 for 150 minutes followed by washing
- Alkaline extraction step of the pulp at 12 % consistency at 85 °C and pH 10.0 for 60 minutes followed by washing
- Second chlorine dioxide bleaching at a pulp consistency of 12 %, temperature 75 °C and pH 3.5-3.9 for 120 minutes followed by washing
- Peroxide step at a pulp consistency of 12 %, temperature 85 °C and pH 10.0 for 90 minutes followed by a final washing

S_{ref2}

- Chlorine dioxide bleaching of a pulp with 10 % consistency at 90 °C and pH 2.7 for 150 minutes followed by washing
- Alkaline extraction step of the pulp at 12 % consistency at 85 °C and pH 11.3 in the presence of 0.2 $MPaO_2$ and peroxide for 60 minutes followed by washing
- Second chlorine dioxide bleaching at a pulp consistency of 12 %, temperature 75 °C, and pH 3.7-3.9 for 120 minutes followed by washing

S_{inv1}

- Chlorine dioxide bleaching of a pulp with 10 % consistency at 90 °C and pH 2.5 for 150 minutes followed by washing
- Alkaline extraction step of the pulp at 12 % consistency at 80 °C and pH 10.5 for 10 minutes followed by addition of chlorine dioxide in order to achieve a chlorine dioxide bleaching at 80 °C for 30 minutes, pH 3.1-3.5
- Addition of peroxide to the pulp in order to achieve a peroxide step at 85 °C and pH 9.5-10 for 90 minutes

S_{inv2}

- Chlorine dioxide bleaching of a pulp with 10 % consistency at 90 °C and pH 2.7 for 180 minutes followed by washing
- Alkaline extraction step of the pulp at 12 % consistency at 80 °C and pH 10.5 for 10 minutes followed by addition of chlorine dioxide in order to achieve a chlorine dioxide bleaching at 80 °C and pH 3.1-3.5 for 30 minutes followed by washing
- Addition of peroxide to the pulp with 12 % consistency in order to achieve a peroxide step at 85 °C and pH 10.0 for 90 minutes

The results show that it is possible to obtain a brightness of 90 % ISO with the sequence S_{inv1} of the invention at approximately the same chemical cost as the reference sequence S_{ref2}. However, the sequence S_{inv1} gives a much lower investment cost of a bleach plant as it requires fewer washing steps. Furthermore, S_{inv1} also provides 0.5 % ISO higher reverted brightness and 20 % lower COD generation than S_{ref2}.

The alternative sequence S_{inv2} according to the invention renders a lower chemical cost. Furthermore, it also provides 0.5 % ISO higher reverted brightness and 15 % lower COD generation than S_{ref2}.

S_{Ref1} has the lowest estimated chemical cost and a slightly higher reverted brightness than the sequence S_{ref2}. The COD generation is also lower than S_{ref2} but the investment cost for this four step sequences is substantially higher than for the sequences according to the invention, S_{inv1} and S_{inv2}, due to the number of washers required.

Table 1

	S_{Ref1}	S_{Ref2}	S_{inv1}	S_{inv2}
Brightness [% ISO]	90	90	90	90
Bleaching stages	4	3	2	3
Total time [min]	420	330	280	280
Washers	4	3	2	3
Bleached pulp				
Rev. brightness [% ISO]	88.0	87.7	88.2	88.2
Viscosity [ml/g]	890	900	840	895
COD total [kg/odt]	24.8	26.1	20.5	22.0
Chemicals				
ClO_2 [kg active Cl]	19	19.5	20.5	21.5
H_2O_2 [kg/odt]	3	3	3	3
NaOH [kg/odt]	8.5	11	11.5	8.5
H_2SO_4 [kg/odt]	3.0	4.0	6.0	5.5
$MgSO_4$ [kg/odt]	1.0	1	1.0	1.0
Oxygen [kg/odt]	-	4.0	-	-
Estimated chemical cost [US$/odt]	14.5	16.5	16.8	15.7

Example 2

A sulphate pulp produced by a wood mixture of 70 % *Eucalyptus nitens* and 30 % *Eucalyptus globulus* was used for laboratory tests. The pulp had, after oxygen delignification (in a processing plant) a kappa number of 8.6, a viscosity of 935 ml/g and a brightness of 64 % ISO. The pulp was bleached according to two sequences according to the invention, S_{inv3} and S_{inv4}, and one reference sequence S_{Ref3}.

The sequences (S_{inv3}, S_{inv4} and S_{Ref3}) are listed below. The results for a brightness of 91 % ISO are shown in Table 2 and the results for a reverted brightness of 89 % ISO are shown in Table 3.

S_{Ref3}

- Chlorine dioxide bleaching of a pulp with 10 % consistency at 90 °C and pH 3.2 for 90 minutes followed by washing
- Alkaline extraction step of the pulp at 12 % consistency at 85 °C and pH 11.3 in the presence of 0.2 MPa O_2 and peroxide during 60 minutes followed by washing
- Second chlorine dioxide bleaching at a pulp consistency of 12 %, temperature 60-75 °C and pH 2.9-3.7 for 120 minutes followed by washing

S_{inv3}

- Chlorine dioxide bleaching of a pulp with 10 % consistency at 90 °C and pH 3.3 for 90 minutes followed by washing
- Alkaline extraction step of the pulp at 12 % consistency at 80 °C and pH 11.4 for 10 minutes followed by addition of chlorine dioxide in order to achieve a chlorine dioxide bleaching at 80 °C and pH 3.0-3.9 for 30 minutes followed by washing
- Addition of peroxide to the pulp with 12 % consistency in order to achieve a peroxide step at 80 °C and pH 11.2-11.5 for 60 minutes

S_{inv4}

- Chlorine dioxide bleaching of a pulp with 10 % consistency at 90 °C and pH 3.3 for 90 minutes followed by washing
- Alkaline extraction step of the pulp at 12 % consistency at 80 °C and pH 11.4 for 10 minutes followed by addition of chlorine dioxide in order to achieve a chlorine dioxide bleaching at 80 °C and pH 3.0-3.9 for 30 minutes followed by washing
- Alkaline extraction step of the pulp at 12 % consistency at 80 °C for 10 minutes followed by addition of chlorine dioxide in order to achieve a chlorine dioxide bleaching at 80 °C and pH 4.9-5 for 60 minutes followed by washing

Table 2

	S_{inv3}	S_{inv4}	S_{Ref3}
Brightness [% ISO]	91	91	91
Bleaching stages	3	3	3
Total time [min]	190	200	270
Washers	3	3	3
Bleached pulp			
Rev brightness [% ISO]	89.0	88.3	88.1
Viscosity [ml/g]	830	820	850
COD total [kg/odt]	17	16	24
Chemicals			
ClO_2 [kg active Cl]	22	28	23
H_2O_2 [kg/odt]	5	-	3
NaOH [kg/odt]	11	10	11
H_2SO_4 [kg/odt]	5	5	5
$MgSO_4$ [kg/odt]	1	0	1
Oxygen [kg/odt]	0	0	4
Estimated chemical cost [US$/odt]	20.3	17.6	19.4

The results show that by utilizing the sequence S_{inv4} it is possible to obtain a brightness of 91 % ISO at a 10 % lower chemical cost and a 30 % lower COD generation than with the reference S_{Ref3}. The sequences S_{inv4} and S_{Ref3} result in substantially the same reverted brightness and will result in approximately the same investment cost of a bleach plant.

The sequence S_{inv3} has a higher chemical cost but the investment cost of a bleach plant will be approximately the same as in the case of the reference S_{Ref3}. However, S_{inv3} results in a 0.9 % higher reverted brightness and a 30 % lower COD generation than the reference S_{Ref3}.

At a reverted brightness of 89 % ISO, the sequences S_{inv3} and S_{inv4} showed 5 % and 12 % lower chemical cost, respectively, when compared to the reference S_{Ref3}.

Table 3

	S_{inv3}	S_{inv4}	S_{Ref3}
Rev. Brightness [% ISO]	89	89	89
Bleaching stages	3	3	3
Total time [min]	190	200	270
Washers	3	3	3
Bleached pulp			
Brightness [% ISO]	91.0	91.5	91.6
Viscosity [ml/g]	835	820	845
Chemicals			
ClO_2 [kg active Cl]	26	35	32
H_2O_2 [kg/odt]	5	-	3
NaOH [kg/odt]	11	10	11
H_2SO_4 [kg/odt]	5	5	5
$MgSO_4$ [kg/odt]	1	0	1
Oxygen [kg/odt]	0	0	4
Estimated chemical cost [US$/odt]	20.3	18.7	21.3

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CLAIMS

1. Method of bleaching a pulp comprising the following steps:
   (i) subjecting an oxygen delignified and washed pulp with a consistency of 8-20 % to a chloride dioxide bleaching step at a temperature of 80-95 °C, preferably 85-95 °C, and a pH of 2-4;
   (ii) washing the pulp;
   (iii) subjecting the washed pulp at a consistency of 8-20 % to an alkaline extraction step at a pH of 8-14, preferably at a pH of 9-12; and
   (iv) adding chlorine dioxide to the alkali containing pulp and adjusting the pH to 2-4 to obtain a second chlorine dioxide bleaching step,
   wherein step (iv) is performed directly after step (iii) without any intermediate wash.
2. Method according to claim 1 wherein step (i) is performed at a pH of 2.5-3.5.
3. Method according to claim 1 or 2 wherein the alkaline extraction step (iii) is performed at a temperature of 75-85 °C.
4. Method according to any of the preceding claims wherein the second chlorine dioxide bleaching step (iv) is performed at a temperature of 75-90 °C.
5. Method according to any of the preceding claims wherein the pulp is subjected to a peroxide treatment directly after, or after an intermediate wash following, the second chlorine dioxide bleaching step (iv).
6. Method according to any of claims 1-4 wherein the pulp is washed after the second chlorine dioxide bleaching step (iv) and subsequently subjected to a second alkaline extraction step at a pH of 8-14 and a third chlorine dioxide bleaching step at a pH of 2-4.
7. Method according to claim 6 wherein the second alkaline extraction step and the third chlorine dioxide bleaching step are performed without any intermediate wash.
8. Method according to any of the preceding claims wherein the pulp is a hardwood pulp.
9. Method according to any of the preceding claims wherein the pulp is a Eucalyptus based pulp.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE2009/050152

A. CLASSIFICATION OF SUBJECT MATTER

IPC: see extra sheet
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: D21C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-INTERNAL, WPI DATA, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	DOMINIQUE LACHENAL ET AL, "High temperature chlorine dioxide delignification: a breakthrough in ECF bleaching of hardwood kraft pulps", Pulping Conference 1998, pages 601-604, abstract, table 3 ---	1-9
Y	WO 9117306 A1 (SUNDS DEFIBRATOR INDUSTRIES AKTIEBOLAG), 14 November 1991 (14.11.1991), page 1, line 11 - line 19; page 3, line 17 - line 20, claim 1 ---	1-9

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
18 May 2009	28-05-2009
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Karin Bengtsson/EÖ Telephone No. +46 8 782 25 00

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE2009/050152

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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International patent classification (IPC)
D21C 9/14 (2006.01)

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Cited literature, if any, will be enclosed in paper form.

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