Multistage brightening of high yield and ultra high-yield wood pulps.

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US-A-2 952 784
US-A-3 100 732


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Description

This invention relates to a method for the brightening of high-yield and ultra high-yield pulps and, more particularly, to a process of super-brightening such pulps with a sequential bleaching process.

Various processes are employed in the pulp and paper industry to bleach high-yield and ultra high-yield pulps. Peroxide is the most commonly employed one stage oxidizing agent for bleaching mechanical pulps. This alkaline process is normally carried out at high pulp consistency, i.e. 15-25%; moderate temperatures—i.e. 60°C; and retention times of 2 to 3 hours. In peroxide bleaching, it is also known to use stabilizers such as sodium silicate and magnesium sulphate in the bleach liquor. This addition prevents decomposition of the oxidizing agent. Furthermore, mechanical pulps are normally pretreated at a low consistency with organic chelating agents, such as sodium diethylenetriamine penta-acetate (DPTA), to remove naturally occurring trace metals. ISO brightnesses of 73-75% are conventionally achieved using this one stage process.

Another one step brightening process for mechanical pulps utilizes sodium hydroxysulfite, a reducing agent, as the brightening agent. This process is usually carried out in an aqueous phase at 3 to 4% consistency, a pH of 4.5 to 6, a temperature of about 60°C and a retention time of one hour. The use of a chelating or sequestering agent such as sodium tripolyphosphate (STPP) is recommended, the agent being added to the pulp prior to the addition of the reducing agent. This method of bleaching usually increases the brightness of mechanical pulps 7-9 ISO-brightness points to a level of 70%.

Two stage bleaching of groundwood pulp using peroxide in the first stage and hydroxysulfite (dithionite) in the second stage is well known and applied commercially. An ISO-brightness level of 76% has been achieved. However, much lower brightness levels are achieved when this two stage sequence is reversed (Schröter, H., Wbl. Papierfabr. 97, No. 23/24 (1969) p. 1023 and Joyce, P. and Mackie, M., CPPA, TAPPI International Pulp Bleaching Conference, Toronto, Canada, June 11-14, 1979, Preprint Page 116). Loras, V and Soteland, N disclose a three stage bleach sequence utilizing in sequence, borohydride, peroxide and dithionite. This sequence was reported to yield a brightness of 88% from an initial level of 67%, an increase of 21 brightness points. (High Brightness Bleaching of Mechanical Pulp, Norsk Skogindustri, 10/72 p. 255). It is also known from U.S. Patent 3,100,732 to Smedberg to use a combined and simultaneous action of an oxidizing agent and a reducing agent; the patentee also discloses that if used in sequence, one uses the oxidizing agent followed by the reducing agent.

There is disclosed in US-A-1 820 502 a method for bleaching pulp as defined in the pre-characterising portion of claim 1 hereof.

An object of the present invention is to provide a method for bleaching high-yield and ultra high-yield pulps by a multistage process providing high brightness levels.

According to the invention there is provided a method for bleaching high-yield and ultra high-yield pulps as defined in the accompanying claims.

In an embodiment of the present invention described below, there is provided a method for the bleaching of high-yield or ultra high-yield pulp which comprises the steps of sequentially treating the wood pulp with a peroxycation compound, a reducing compound and a second peroxycation compound.

In greater detail, the method or process includes at least three stages wherein the wood pulp is subjected to bleaching operations. The wood pulp which may be utilized is any high-yield or ultra high-yield pulp such as mechanical, chemimechanical, chemithermomechanical, groundwood and high-yield sulphite pulps.

In the first stage, the pulp is bleached with a peroxycation compound. Preferred conditions of bleaching include:

(1) a peroxycation compound charge of about 0.5 to about 3% by weight of oven dried pulp in the presence of sodium hydroxide, sodium silicate and magnesium sulphate
(2) a reaction temperature of between about 60°C to about 100°C;
(3) a reaction time of from about 4 minutes to about 120 minutes;
(4) a pulp consistency of from about 6% to about 25%; and
(5) a reaction terminating pH of from about 7.5 to about 9.5.

Subsequent to the first bleaching step, one may utilize an optional washing step. This washing step is not necessary; if the washing step or treatment is utilized, a compound such as SO₃⁻H₂ ions in aqueous solution may be utilized to bring the pH into a range of from about 5 to 6.

In the second stage, the wood pulp is treated with a reducing compound which may be chosen from many such reducing compounds known to those skilled in the art. During this second stage, preferred reaction conditions include:

(1) a reducing compound charge of about 0.1 to about 3.0% by weight of oven dried pulp;
(2) the presence of a chelating agent such as DPTA or STPP;
(3) a reaction temperature of from about 60°C to about 100°C;
(4) a reaction time of from about 4 to about 120 minutes;
(5) a pulp consistency of from about 3% to about 25%, and
(6) a reaction terminating pH of about 3.5 to about 10.5.

After the above reduction step, a washing or pressing step as is known in the art is subsequently carried out. The third and final bleaching stage utilizes a
peroxxygen compound as in the first stage. The preferred reaction conditions include:

1. a peroxxygen compound charge of about 0.01% to about 2.0% in the presence of sodium hydroxide, sodium silicate and magnesium sulphate;
2. a reaction temperature of from about 60°C to about 100°C;
3. a reaction time of about 4 minutes to about 240 minutes;
4. a pulp consistency of from about 6% to about 25%; and
5. a reaction terminating pH from about 7.5 to about 9.5.

The compounds utilized in the process of the present invention may be selected from among those well known to those skilled in the art. Thus, the peroxxygen compound utilized in the first and third stages may include conventional inorganic peroxides such as hydrogen and sodium peroxide and also organic peroxides such as benzoyl peroxide, dibenzoyl peroxide and peroxyacids. Examples of reducing compounds which may be chosen from commercially inorganic reducing agents such as sodium or zinc hydrosulphite (dithionite), sodium as magnesium bisulphite, sodium borohydride, borof®, thiourea dioxide, ammonium borohydride, hydrazine and organic reducing agents such as amine-boranes and phoshine-boranes. It will be noted that some of these reducing agents are sold commercially with a chelating agent mixed therewith. However, when a chelating agent has already been introduced with the pulp prior to the first stage peroxide treatment, as is a common practice, no further addition of a chelating compound is required.

The most preferred charge of the peroxxygen compound in the first stage is 1—2.5% by weight of oven-dried pulp. Sodium hydroxide, sodium silicate and magnesium sulphate are preferably added in charge ranges of 0.25—2.0, 0.0 to 2.5% and 0.01—0.05%, respectively, in order to stabilize the peroxxygen compound, in the form of the peroxyhydroxyl ion and to initiate and maintain a stable bleaching reaction. Although it is common practice in a one-stage bleaching process to leave about 15 to 20% of the initial charge of peroxxygen compound as a residual to prevent alkali darkening of the pulp, because of a shorter reaction time, it is preferred to lower this residual level to 1 to 10% in order to conserve SO₃⁻₂ and allow a lower initial charge of oxidant addition. Contrary to present practice, a temperature range of 75 to 90°C and a 10 to 20 minute reaction time is preferred for optimized bleaching efficiency in the first stage. The higher temperature together with efficient mixing allows the use of a short reaction time while achieving maximum brightness levels. Also, with proper attention to efficient mixing, a pulp consistency of about 10% was found to be as effective as a 30% pulp consistency for brightness improvement.

In the second stage, it has been found that the maximum brightness level is achieved utilizing a charge of about 0.3% to about 3.0% by weight of the reducing agent based on oven-dried pulp. With efficient mixing, for maximum brightness, the most preferred temperature range is between 65°C to 85°C; a consistency of between 4% and 16%; and a reaction time of between 1 to 20 minutes.

In the third stage, wherein the wood pulp is treated with a second peroxxygen compound, the preferred charge of oxidant is between 0.3% to 2.0% by weight based on O.D. wood. The charges of sodium hydroxide, sodium silicate and magnesium sulphate, by weight are between 0.1 to 1.0%, 0.0—2.0% and 0.01%—0.05% by weight respectively. The most preferred ranges of temperature, time and pulp consistency are respectively 75°C—95°C, 10—60 minutes and 9%—20%. After the bleaching, the pulp is subjected to a pressing and/or washing step or treated with a compound such as sulphur dioxide which contains SO₃⁻ ions in aqueous solution to bring the pH to about 6. However, if there is residual peroxxygen remaining after the final reaction it can be separated from the pulp, i.e. by pressing and the pressate recirculated to the first and/or third stages as part of the make-up solution.

As an optional step, and prior to the first stage wherein the pulp is bleached with a peroxxygen compound, the pulp may be subjected to a pre-treatment step with a reducing compound. In this pre-treatment step, one may utilize a charge of between about 0.1% to 3.0% by weight of the reducing agent based on oven dried pulp. A preferred temperature range is between 40°C and 100°C; a consistency of between 3% and 25% is preferred with a preferred reaction time of between 1 and 60 minutes; and at reaction terminating pH of between 7 and 12. The pre treated pulp may then be pressed to the desired consistency for the first stage. The compounds employed may be selected from those previously discussed.

Having thus generally described the invention, reference will be made to the following examples.

Example 1

100 g of commercial spruce balsam chemithermomechanical pulp, brightness 56.1% ISO taken from a disc filter of a mill was treated with a solution containing 2.0% hydrogen peroxide and O.D. pulp, 2.5% sodium silicate, 1.0% sodium hydroxide, 0.05% magnesium sulphate with sufficient water to reduce the pulp consistency to 10%. The temperature of this treatment was 85°C and the reaction time was 10 minutes. At the end of the reaction the pH was 8.5. The residual peroxide concentration was tested and found to be 0.4% H₂O₂ on O.D. pulp. The brightness of a washed sample after this stage was 70% ISO. A portion of the peroxide treated pulp was treated with SO₂ bringing the pH to 6 and a solution containing 0.8% sodium hydrosulphite on O.D. pulp and 0.2% chelating agent STPP was added to the pulp with enough water to bring the con-
sistency to 10%. The temperature of the reaction was 75°C and the reaction time was 15 minutes. At the end of the reaction the pH of the pulp and the bleaching solution was 5.1. The pulp was carefully washed by bringing the pulp down from 10% consistency to 1% and then raised to 14%. The brightness of the pulp at the completion of the stage was 79.6% ISO.

To complete the three stage process, the peroxide/hydrosulphite pulp was treated with a solution containing 2.0% hydrogen peroxide on O.D. pulp, 2.5% sodium silicate, 1.0% sodium hydroxide and 0.05% magnesium sulphate with sufficient water to reduce the pulp consistency to 10%. The reaction temperature was 65°C and the reaction time was 10 minutes. The pH at the end of the reaction was 8.2 and the pulp after souring and washing had a brightness of 84.2%. The peroxide residual measured at the end of the reaction was 1.6% H₂O₂ on O.D. pulp. In subsequent experiments, this residual was separated from the pulp by pressing and the pressate was recirculated to the first and/or third bleaching stages. No detrimental effect on the final bleaching properties of the pulp was observed.

Example II
A comparative test using conventional conditions for each stage of a three stage bleaching sequence was conducted on the same sample of the unbleached pulp used in Example I. 100 g of the pulp was treated with a solution containing 2% hydrogen peroxide, 2.5% sodium hydroxide, 2.5% sodium silicate, 0.05% magnesium sulphate and sufficient water to reduce the pulp consistency to 10%. The pulp was then placed into a bath and the temperature maintained at 60°C for 2 hours. A sample of pulp tested for brightness after souring with SO₂ and washing showed the pulp had reached a brightness of 70% ISO. The residual peroxide concentration measured at this point was equivalent to 0.3% H₂O₂ on pulp O.D. basis. A portion of the peroxide treated pulp was soured with SO₂ to pH 6 and then treated with a 0.83% solution of sodium hydrosulphite including 1.5% of STPP and enough water to bring the consistency to 5%. The reaction temperature was 60°C and the retention time was 1 hour. A portion of the pulp was washed and the brightness measured on this sample was 74.5% ISO.

The washed peroxide/hydrosulphite pulp was then treated in a second peroxide oxidation stage using a 2.0% solution containing hydrogen peroxide on O.D. pulp, 2.5% sodium silicate, 2.5% sodium hydroxide, 0.05% magnesium sulphate and sufficient water to reduce the pulp consistency to 15%. The temperature of the pulp was maintained at 60°C for 2 hours. The final pH at the end of this period was 9.5. The pulp was then soured with SO₂ to pH 6 and washed. The final brightness of the peroxide hydrosulphite peroxide pulp was 79.4% ISO. The residual peroxide concentration was measured to be 1.1% H₂O₂ on O.D. pulp.

Example III
100 g of commercial spruce balsam thermomechanical pulp, brightness 59.7% ISO was treated with a solution containing 2% hydrogen peroxide on O.D. pulp, 2.0% sodium silicate, 1.8% sodium hydroxide, 0.05% magnesium sulphate, and enough water to reduce the pulp consistency to 25%. The temperature of this treatment was 83°C. Treatment time was 25 minutes. The residual hydrogen peroxide charge was tested and found to be 0.6% on O.D. pulp. The brightness after this stage was 71.2% ISO. The peroxide treated pulp was treated with sodium bisulphite bringing the pH to 6. The excess sodium bisulphite was washed out with water and a solution containing 0.5% sodium borohydride and 0.6% sodium hydrosulphite was added to the pulp along with enough water to bring the consistency to 15%. The temperature of this treatment was 60°C and the treatment time was 30 minutes. The brightness of the pulp at this stage was 72.4% ISO.

A portion of the peroxide/borohydride treated pulp was pressed to consistency greater than 25% and treated with a 2.0 solution of hydrogen-peroxide, 2.0% sodium silicate (as 41° Be solution) on O.D. pulp, 2.0% sodium hydroxide, and 0.05% magnesium sulphate, and sufficient water to reduce the consistency to 25%. This third stage treatment was conducted at a temperature of 80°C for a time of 40 minutes. The hydrogen peroxide residual was 0.64% charge on O.D. pulp. After washing with a sodium bisulphite solution, the pulp brightness achieved was 80.5% ISO.

Example IV
100 g of a commercial western spruce-lobolly pine chemithermomechanical pulp, brightness 56.8% ISO taken from the latency chest of a mill was treated with a solution containing 2.0% hydrogen peroxide on O.D. pulp, 2.5% sodium silicate, 2.0% sodium hydroxide, 0.05% magnesium sulphate with sufficient water to reduce the pulp consistency to 10%. The temperature of this treatment was 90°C and the reaction time was 10 minutes. At the end of the reaction the pH was 8.9. The residual peroxide concentration was tested and found to be 0.3% H₂O₂ on O.D. pulp. The brightness of a washed sample after this stage was 72% ISO. A portion of the peroxide treated pulp was treated with SO₂ bringing the pH to 6 and a solution containing 0.83% sodium hydrosulphite on O.D. pulp and 0.2% chelating agent STPP was added to the pulp with enough water to bring the consistency to 10%. The temperature of the reaction was 85°C and the reaction time was 11 minutes. At the end of the reaction the pH of the pulp and the bleaching solution was 5.6. The pulp was carefully washed by bringing the pulp down from 10% consistency to 1% and then raised to 14%. The brightness of the pulp at the completion of the stage was 77.3% ISO.

To complete the three stage process, the peroxide/hydrosulphite pulp was treated with a sol-
olution containing 2.0% hydrogen peroxide on O.D. pulp, 2.5% sodium silicate, 2.0% sodium hydroxide and 0.05% magnesium sulphate with sufficient water to reduce the pulp consistency to 10%. The reaction temperature was 90°C and the reaction time was 10 minutes. The pH at the end of the reaction was 8.8 and the pulp after souring and washing had a brightness of 83.6%. The peroxide residual measured at the end of the reaction was 1.8% H₂O₂ on O.D. pulp.

Example V
100 g of a commercial spruce-balsam high yield sulphite pulp, brightness 58.2% ISO taken from the high density storage of a mill was treated with a solution containing 2.0% hydrogen peroxide on O.D. pulp, 2.5%, sodium silicate, 2.0% sodium hydroxide, 0.05% magnesium sulphate with sufficient water to reduce the pulp consistency to 10%. The temperature of this treatment was 85°C and the reaction time was 10 minutes. At the end of the reaction the pH was 8.9. The residual peroxide concentration was tested and found to be 0.1% H₂O₂ on O.D. pulp. The brightness of a washed sample after this stage was 66.7% ISO. A portion of the peroxide treated pulp was treated with SO₂ bringing the pH to 6 and a solution containing 0.8% sodium hydroxosulphite on O.D. pulp and 0.2% chelating agent STPP was added to the pulp with enough water to bring the consistency to 10%. The temperature of the reaction was 80°C and the reaction time was 15 minutes. At the end of the reaction the pH of the pulp and the bleaching solution was 5.0. The pulp was carefully washed by bringing the pulp down from 10% consistency to 1% and then raised to 14%. The brightness of the pulp at the completion of the stage was 72.6% ISO.

To complete the three stage process, the peroxide/hydroxosulphite pulp was treated with a solution containing 2.0% hydrogen peroxide on O.D. pulp, 2.5% sodium silicate, 2.0% sodium hydroxide and 0.05% magnesium sulphate with sufficient water to reduce the pulp consistency to 10%. The reaction temperature was 90°C and the reaction time was 20 minutes. The pH at the end of the reaction was 9.1 and the pulp after souring and washing had a brightness of 81.3%. The peroxide residual measured at the end of the reaction was 1.3% H₂O₂ on O.D. pulp.

Example VI
100 g of commercial spruce-balsam chemithermomechanical pulp, brightness 53.7% ISO taken from the second stage refiner at 24% pulp consistency was diluted with enough mill white water containing 0.2% DTPA on O.D. pulp, to reduce the consistency of the pulp to 4%. The pulp was then drained to 15% consistency. The pulp was treated with a solution containing 2% hydrogen on O.D. pulp, 1.75% sodium hydroxide, 2.5% sodium silicate, 0.05% magnesium sulphate and sufficient water to reduce the pulp consistency to 10%. The temperature of this treatment was 85°C and the reaction time was 30 minutes. At the end of this reaction the pH was 8.7. The pulp stock was pressed to 27% consistency. The residual peroxide concentration was determined and found to be 0.97% H₂O₂ on O.D. pulp.

The brightness of a sample taken from the pressed stock after this stage was 70.3% ISO. The remaining pressed peroxide treated stock was treated with a solution containing 0.3% BOROL® on O.D. pulp (equivalent to 0.043% sodium boroxydride on O.D. pulp) with sufficient water to reduce the pulp consistency to 10%. The temperature of this treatment was 65°C and the reaction time was 10 minutes. At the end of reaction the pH was 10.1. The pulp stock was pressed to 27% consistency. The brightness of a sample taken from the peroxide/BOROL® treated pulp was 71.7% ISO.

To complete the three-stage process, one-half of the peroxide/BOROL® treated pulp was further treated with a solution containing 2% hydrogen peroxide on O.D. pulp, 2.0% sodium hydroxide, 2.5% sodium silicate, 0.05% magnesium sulphate with sufficient water to reduce the pulp consistency to 10%. The reaction temperature was 88°C and the time was 120 minutes. The pH at the end of the reaction was 9.9 and the pulp after washing had a brightness of 81.7% ISO. The peroxide residual measured at the end of the reaction was 1.1%. In subsequent experiments, this residual was separated from the pulp by pressing and the pressate was recirculated to the first and/or third bleaching stage.

To the other half of the peroxide/BOROL® treated pulp, a solution containing 2.0% hydrogen peroxide on O.D. pulp, 2% sodium hydroxide, 2.5% sodium silicate and 0.05% magnesium sulphate with sufficient water to reduce the pulp consistency to 20%. The reaction temperature was 83°C and the retention time 120 minutes. The pH at the end of the reaction was 10.2 and the pulp after washing had a brightness of 82.5% ISO. The peroxide residual at the end of the reaction was 0.62% on pulp O.D. basis.

As will be seen from the above, the novel process of the present invention provides pulp with a high brightness. Example II, which used the same sequence as Example I, but conventional conditions as taught by the art, did not achieve the same brightness.

Example VII
100 g of commercial spruce-balsam chemithermomechanical pulp, brightness 53.7% ISO taken from the second stage refiner at 24% pulp consistency was treated with a solution containing 0.3% BOROL® on O.D. pulp (equivalent to 0.043% sodium boroxydride on pulp) and 0.2% DTPA on pulp with sufficient water to reduce the consistency to 10%. The temperature of this pretreatment was 70°C and the time was 15 minutes. At the end of the reaction, the pH was 9.5. The pulp stock was pressed to 15% consistency.

The pulp was then treated with a solution containing 2.0% hydrogen peroxide on O.D. pulp, 1.75% sodium hydroxide, 2.5% sodium silicate,
0.05% magnesium sulphate and sufficient water to reduce the pulp consistency to 10%. The temperature of this treatment was 84°C and the reaction time was 30 minutes. At the end of this reaction, the pH was 9.5. The pulp stock was pressed to 27% consistency. The residual peroxide concentration was found to be 1.04%. The brightness of a sample taken from the pressed stock, after this stage, was 71.4% ISO. The remaining pressed BOROL®/peroxide treated stock was treated with a solution containing 0.3% BOROL® on O.D. pulp (equivalent to 0.043% sodium borohydride on O.D. pulp) with sufficient water to reduce the consistency to 10%. The temperature of this treatment was 65°C and the reaction time, 10 minutes. At the end of the reaction, the pH was 10.3. The brightness of a sample taken from the BOROL®/peroxide/BOROL® treated pulp was 72.8% ISO.

To complete the four-stage process, the BOROL®/peroxide/BOROL® treated pulp was further treated with a solution containing 2% hydrogen peroxide on O.D. pulp, 2.0% sodium hydroxide, 2.5% sodium silicate, 0.05% magnesium sulphate with sufficient water to reduce the pulp to 10%. The reaction temperature was 80°C and the time was 120 minutes. The pH at the end of the reaction was 10.3 and the pulp after washing had a brightness of 81.6% ISO. The peroxide residual measured at the end of the reaction was 1.13%.

Example VIII
Spruce chemithermomechanical pulp, taken from the second stage refiner at 20 percent consistency was diluted to 1 per cent consistency, and DTPA at a charge of 0.15 per cent on oven-dried O.D. pulp was added. The pulp slurry was stirred at 60°C for 1 hour and then filtered to 20% consistency. The ISO brightness after this washing stage was 60.1%.

The pulp was then pretreated with BOROL® solution at a charge of 0.275% on O.D. pulp and DTPA at a charge of 0.15% with sufficient water to decrease the consistency to 15%. The temperature of the treatment was 80°C and the time was 15 minutes. At the end of the pretreatment the pH was 8.4.

The pulp stock was pressed to 30% consistency and treated with a charge of 2% hydrogen peroxide, 2.5% sodium hydroxide, 2.2% sodium silicate and 0.05% magnesium sulphate. All charges are based on dry weight of pulp. Sufficient water was used to bring the consistency to 25%. The temperature of hydrogen peroxide treatment (P1 stage) was 80°C and the time of treatment was 90 minutes. At the end of this treatment the pH was 9.8 and the hydrogen peroxide residual was 0.18% on O.D. pulp. The ISO brightness was 73.5%.

The pulp was treated with a BOROL® in a reduction stage at a charge of 0.275% and DTPA at a charge of 0.15% on O.D. pulp with sufficient water to decrease the consistency to 15%. The temperature of the treatment was 80°C and the time was 15 minutes. At the end of the BOROL® treatment (R stage) the pH was 10.0.

After the BOROL® treatment, the pulp was washed by diluting to 1% consistency and then dewatering to 30% consistency by filtration and pressing.

The process was completed by treating the BOROL®/peroxide/BOROL® pulp with 2% hydrogen peroxide on O.D. pulp, 1.5% sodium hydroxide, 2.2% sodium silicate, and 0.06% magnesium sulphate with enough water to bring the consistency to 25%. The treatment temperature was 80°C and the time was 240 minutes. The pH at the end of the third stage was 10.0 and the residual hydrogen peroxide was 0.65% on O.D. pulp. After washing, the pulp had an ISO brightness of 81.4%.

Claims
1. A method for bleaching pulp comprising two bleaching steps and an intermediate step of treatment with a reducing agent characterised in that said pulp is a high-yield or ultra high-yield pulp and said two bleaching stages each comprise treatment of said pulp with a peroxide compound and said second treatment of the pulp with a peroxide compound produces a further increase in brightness of the pulp after said intermediate step of treatment with a reducing agent.

2. The method of claim 1 characterized by:
   (i) the first treatment with said peroxide compound being at a charge of between 0.5 to 3% by weight based on oven dried pulp at a temperature of between 60°C to 100°C for a period of from 4 minutes to 120 minutes at a pulp consistency of between 6% and 25% and a reaction terminating pH of from 7.5 to 9.5;
   (ii) the treatment of the pulp with the reducing agent being at a charge of 0.1 to 3.0% by weight in the presence of a chelating agent at a reaction temperature of between 60°C to 100°C for a time of between 4 to 120 minutes at a pulp consistency of from 3% to 25% and a reaction terminating pH of between 3.5 to 10.5;
   the process being further characterized by washing or pressing said pulp after said treatment with a reducing agent and subsequently treating said washed pulp with a second peroxide compound at a charge of between 0.01% to 2.0% by weight at a temperature of between 60°C to 100°C for a time period of between 4 minutes to 240 minutes at a pulp consistency of between 6% to 25% and a reaction terminating pH of between 7.5 to 9.5.
3. The method of any one of the preceding claims characterized by the treatment of the pulp with the first and second peroxide compounds being done in the presence of sodium hydroxide, sodium silicate and magnesium sulphate.

4. The method of any one of the preceding claims characterized by the step of subsequently treating the pulp following the second peroxide treatment with a washing step or treating with sulphur dioxide to bring the pH to 6.

5. The method of any one of the preceding claims characterized by the peroxide compounds employed in said bleaching steps being
selected from the group consisting of hydrogen peroxide, sodium peroxide, benzyl peroxide, diteriarybutyl peroxide and peracetic acid.

6. The method of any one of the preceding claims characterized by the reducing agent being selected from the group consisting of sodium hydroxysulphite, sodium bisulphite, magnesium bisulphite, zinc hydroxyhypochlorite, sodium borohydride, thiourea dioxide, ammonium borohydride and hydrazine.

7. The method according to any one of the preceding claims characterized by the pulp being selected from the group of pulps consisting of mechanical, chemimechanical, chemithermomechanical, groundwood and high-yield sulphite pulps.

8. The method of any one of the preceding claims characterized by the step of pretreating the pulp with a reducing agent at a charge of between 0.1 to 3.0% by weight at a reaction temperature of between 40°C and 100°C for a time of between 1 and 60 minutes at a pulp consistency of from 3 to 25% with a reaction terminating pH of between 7 and 12.

Patentansprüche


2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß man:
   (i) die erste Behandlung mit dieser Peroxidverbindung in einer Menge von zwischen 0,5 und 3 Gew.-% bezogen auf trockenen Stoff, bei einer Temperatur von zwischen 60°C und 100°C für eine Zeitspanne von 4 min bis 120 min bei einer Stoffdichte von 6% und 25% und einem Reaktionend-pH von 7,5 bis 9,5 durchführt;
   (ii) die Behandlung des Stoffes mit dem Reduktionsmittel in einer Menge von 0,1 bis 3,0 Gew.-% in Gegenwart eines chelatbildenden Mittels bei einer Reaktionstemperatur von zwischen 60°C und 100°C für eine Zeitspanne zwischen 4 min und 120 min bei einer Stoffdichte von 3% bis 25% und einem Reaktionend-pH zwischen 3,5 und 10,5 durchführt;
   wobei sich das Verfahren weiter dadurch auszeichnet, daß man diesen Stoff nach dieser Behandlung mit einem Reduktionsmittel wäscht oder preßt und anschließend diesen gewaschenen Stoff mit einer zweiten Peroxidverbindung in einer Menge von 0,01 bis 2,0 Gew.-% bei einer Temperatur zwischen 60°C und 100°C für eine Zeitspanne zwischen 4 min und 240 min bei einer Stoffdichte von 6% und 25% und einem Reaktionend-pH zwischen 7,5 und 9,5 behandelt.


4. Verfahren nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß anschließend nach der zweiten Peroxidbehandlung der Stoff durch eine Waschstufe behandelt oder mit Schwefeldioxid, um das pH auf 6 zu bringen, behandelt wird.


8. Verfahren nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß der Stoff mit einem Reduktionsmittel in einer Menge von zwischen 0,1 und 3 Gew.-% bei einer Reaktionstemperatur zwischen 40°C und 100°C für eine Zeitspanne zwischen 1 und 60 min bei einer Stoffdichte von 3 bis 25% mit einem Reaktionend-pH zwischen 7 und 12 vorbehandelt wird.

Revendications

1. Procédé pour blanchir la pâte comprenant deux stades de blanchiment et un stade intermédiaire de traitement avec un agent réducteur, caractérisé en ce que ladite pâte est une pâte à haut rendement ou à très haut rendement et lesdits deux stades de blanchiment comprennent chacun le traitement de ladite pâte avec un composé péroxygéné et ledit second traitement de la pâte avec un composé péroxygéné produit un accroissement complémentaire du brillant de la pâte après ledit stade intermédiaire de traitement avec un agent réducteur.

2. Procédé selon la revendication 1, caractérisé en ce que
   (i) le premier traitement avec ledit composé péroxygéné est effectué avec une teneur (en anglais: "charge") en composé péroxygéné comprise entre 0,5 et 3% en poids, par rapport au poids de pâte séché à l'étuve, à une température comprise entre 60°C et 100°C, pendant une période de 4 minutes à 120 minutes, à une concentration de la pâte comprise entre 6% et 25% et un pH de fin de réaction de 7,5 à 9,5;
   (ii) le traitement de la pâte avec l'agent réducteur...
est effectué avec une teneur en agent réducteur de 0,1 à 3,0% en poids, en présence d'un agent chélant, à une température de réaction comprise entre 60°C et 100°C, pendant une durée de 4 à 120 minutes, à une concentration de la pâte de 3% à 25% et un pH de fin de réaction de 3,5 à 10,5.

Ledit procédé étant de plus caractérisé par le lavage ou le pressage de ladite pâte après ledit traitement avec un agent réducteur et le traitement suivant de ladite pâte lavée avec un second composé péroxygéné à une teneur comprise entre 0,01% et 2,0% en poids, à une température entre 60°C et 100°C, pendant une durée comprise entre 4 minutes et 240 minutes, à une concentration de la pâte comprise entre 6% et 25% ut un pH de fin de réaction de 7,5 à 9,5.

3. Procédé selon l'une quelconque des revendications précédentes, caractérisé en ce que le traitement de la pâte avec le premier et le second composés péroxygénés est effectué en présence d'hydroxyde de sodium, de silicate de sodium et de sulfate de magnésium.

4. Procédé selon l'une quelconque des revendications précédentes, caractérisé par le stade de traitement ultérieur de la pâte, résultant du second traitement avec un composé péroxygéné, comprenant un stade de lavage ou un traitement avec du dioxyde de soufre pour porter le pH à 6.

5. Procédé selon l'une quelconque des revendications précédentes, caractérisé en ce que les composés péroxygénés utilisés dans lesdits stades de blanchiment sont choisis parmi l'ensemble constitué par le peroxide d'hydrogène, le peroxyde de sodium, le peroxyde de benzyle, le peroxyde de di-tert-butyle et l'acide peracétique.

6. Procédé selon l'une quelconque des revendications précédentes, caractérisé en ce que l'agent réducteur est choisi parmi l'ensemble constitué par l'hydroxyde de sodium, le bisulfite de sodium, le bisulfite de magnésium, l'hydroxyde de zinc, le borohydure de sodium, le dioxyde de thourée, le borohydure d'ammonium et l'hydrazine.

7. Procédé selon l'une quelconque des revendications précédentes, caractérisé en ce que le pâte est choisie parmi l'ensemble des pâtes constitué par les pâtes mécaniques, les pâtes chimiomécaniques, les pâtes chimiothermopécaniques, les pâtes de bois broyé et les pâtes dites au sulfite à haut rendement.

8. Procédé selon l'une quelconque des revendications précédentes, caractérisé par le stade de prétreatment de la pâte avec un agent réducteur à une teneur comprise entre 0,1 et 3,0% en poids, à une température de réaction entre 40°C et 100°C, pendant une durée comprise entre 1 et 60 minutes, à une concentration de la pâte de 3 à 25% et un pH de fin de réaction compris entre 7 et 12.