



Europäisches Patentamt
European Patent Office
Office européen des brevets

Publication number:

**0 191 756
B1**

(12)

EUROPEAN PATENT SPECIFICATION

- (45) Date of publication of patent specification: **04.01.89** (51) Int. Cl.⁴: **D 21 C 9/10**
(21) Application number: **86890030.9**
(22) Date of filing: **12.02.86**

(54) **Multi peroxide stage mechanical pulp bleaching.**

(30) Priority: **15.02.85 US 702000**

(43) Date of publication of application:
20.08.86 Bulletin 86/34

(45) Publication of the grant of the patent:
04.01.89 Bulletin 89/01

(84) Designated Contracting States:
AT DE FR

(56) References cited:
EP-A-0 187 477
US-A-1 820 502
US-A-2 290 601
US-A-2 862 784

**TAPPI, vol. 53, no. 12, December 1970, pages
2275-2277, Atlanta, GA, US; C.A. RICHARDSON
et al.: "Two-stage bleaching of high-yield pulp
at blandin"**

(73) Proprietor: **KAMYR AB**
Box 1033
-S-651 15 Karlstad (SE)

(72) Inventor: **Tibbling, Petter**
c/o Kamy AB Box 1033
S-651 15 Karlstad (SE)

(74) Representative: **Haffner, Thomas M., Dr. et al**
Patentanwaltskanzlei Dipl.-Ing. Adolf
Kretschmer Dr. Thomas M. Haffner
Schottengasse 3a
A-1014 Wien (AT)

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European patent convention).

Courier Press, Leamington Spa, England.

EP 0 191 756 B1

Description

The invention relates to a method of obtaining high brightness lignocellulose pulp comprising the steps of subjecting a mechanical lignocellulose pulp to peroxide bleaching and a hydrosulfite bleaching sequence, and to a high brightness mechanical lignocellulose pulp.

Typical conventional bleaching sequences for the bleaching of mechanical pulps, such as mechanical pulp, chemimechanical pulp (CMP), thermomechanical pulp (TMP) and chemithermomechanical pulp (CTMP) comprise peroxide (P), hydrosulfite (T), or P-T sequences.

Conventional peroxide bleaching, with sodium or hydrogen peroxide, is typically accomplished at a consistency of about 12—30%. Single stage peroxide treatment is most common. However, it has been known to use two stage peroxide bleaching (P-P) for medium brightness mechanical pulps, that is having a brightness of about 75—80% ISO.

Reductive bleaching with hydrosulfites (dithionites) is also commonly practiced in a single stage. However it has been known to use a P-T bleaching sequence, which gives brightness in the range of about 74—78% ISO. In a P-T sequence, the contribution to brightness of the final T stage is gradually reduced as the charge of peroxide is increased in order to boost final brightness.

From EP—A—0 187 477 a multistage process for bleaching lignocellulose pulp is known whereby the pulp is treated sequentially with a peroxygen compound, a reducing compound, e.g. sodium hydrosulfite, and a final peroxygen compound to achieve higher brightness levels, i.e. a P-T-P sequence.

The invention as claimed is intended to further improve the result of treatment without adding further steps to the process.

According to the present invention it has been surprisingly found that where the peroxide treatment is split into two, or more, stages, the T stage does produce a substantial brightening effect, on the order of 2—4 ISO units. In a typical P-T sequence, the contribution from the hydrosulfite stage is very small (less than 2 ISO units) at the 80 + ISO level.

According to the present invention the method is characterized by subjecting the mechanical pulp to a first peroxide bleaching, and then to a second peroxide bleaching, and then to the hydrosulfite bleaching sequence, to produce a bleached mechanical pulp. More than two peroxide stages may be provided, and the sequence of steps preferably practiced sequentially. Preferably the pulp is pressed between the stages to partially remove interstitial pulp liquor. This removed liquor (filtrate) is normally reused for dilution in preceding stages, or for chemical recovery. During the practice of all of steps the pulp maintains a consistency of between 8—50%, although after pressing it is typically diluted (e.g. to between 8—15%). During each actual bleaching stage the consistency is between 8—30%.

The invention also relates to a mechanical pulp

having high brightness, and produced by the practice of the aforementioned sequence of steps. According to the invention, a mechanical pulp is produced having a brightness of 83% ISO (or greater).

It is the primary object of the present invention is produce a high brightness mechanical pulp. This and other objects of the invention will become clear from a description of the detailed description of the invention, and from the appended claims.

Brief description of the drawings

Figure 1 is a schematic view illustrating the steps in the practice of an exemplary method according to the present invention;

Figure 2 is a schematic indicating a preferred modification of the method of Figure 1;

Figure 3 is a schematic representation of exemplary apparatus utilized in the practice of one of the peroxide stages in the method of Figure 1;

Figure 4 is a schematic representation of exemplary equipment utilizable in the practice of the hydrosulfite bleaching stage of Figure 1; and

Figure 5 is a schematic representation of a modification of the method of Figure 1 wherein the T stage is between two P stages.

Detailed description of the drawings

The invention is useful in increasing the brightness of all types of mechanical pulps. The term "a mechanical lignocellulose pulp" as used in the specification and claims is intended to encompass conventional mechanical pulps, such as ground wood and refiner pulps, e.g. CMP, CTMP, and TMP. Figure 1 illustrates the production of a mechanical pulp in a typical mechanical pulping process station 10, which includes the use of refiners, and other pulping process stages as desired.

After a mechanical lignocellulose pulp is produced in station 10, it is subjected to a first sodium or hydrogen peroxide bleaching P_1 , as indicated by reference numeral 12 in Figure 1. Peroxide bleaching is accomplished utilizing conventional equipment, and conventional bleaching conditions for peroxide bleaching of mechanical pulps (e.g. 45—90°C, 15—240 minutes). A typical form such equipment can take is illustrated schematically in Figure 3 wherein the pulp is fed to a mixer 14, the peroxide, sodium hydroxide, and buffering and stabilizing agents or the like (such as sodium silicate and $MgSO_4$) are added. If desired after mixing in mixer 14, the consistency of the pulp may be increased in screw press 15, and ultimately the pulp is fed to bleaching tower 16. Bleaching would typically be accomplished at a consistency of about 8—30%, preferably about 12—25%.

In the present embodiment after treatment in stage P_1 , the pulp is passed to the second peroxide stage P_2 , indicated by reference numeral 18 in Figure 1. There it is subjected to another typical peroxide bleaching sequence. Other peroxide bleaching sequences after stage 12 (e.g. P_3 , P_4 , etc.) may be provided.

In the preferred embodiment of the invention, between the P_1 and P_2 stages the mechanical pulp

is pressed. This is illustrated schematically in Figure 2 wherein a press station 19 is disposed between the peroxide bleaching sequence stages 12, 18. In press station 19, the pulp is pressed to a high consistency (e.g. on the order of 30—50%) before the bleach liquid for the subsequent peroxide stage 18 is added. Also as indicated in Figure 2, pressing in press station 19' preferably is practiced after stage 18.

After the second (or subsequent, i.e. last) peroxide bleaching sequence, the pulp is subjected to a hydrosulfite bleaching, as illustrated schematically by reference numeral 20 in Figure 1. The conditions in the hydrosulfite bleaching stage 20 are conventional (e.g. 45—90°C, 10—60 min.) except that it is preferred that the bleaching be practiced with a pulp consistency of between about 8—30%, preferably between about 8—15%. Hydrosulfite bleaching of medium consistency mechanical pulps is illustrated in EP—A—0,155,928, and the equipment and processes illustrated therein may be utilized in the practice of bleaching 20. Figure 4 schematically illustrates one form of the typical equipment that could be utilized in the practice of stage 20, wherein the pulp, after having been pressed and diluted to about 8—15% consistency, is mixed with hydrosulfite and other chemicals, such as buffering, chelating, and sequestering agents. The pulp is fluidized by the mixer 22, and then passes to conventional upflow tower 24.

Between stations 18, 20, it is also desirable to reduce the pH (e.g. to between 5.5—6.0) of the mechanical pulp. This is typically accomplished by the introduction of SO₂ water. Where the addition of the SO₂ water will dilute pulp consistency below the desired level for station 20, pressing of the pulp can be practiced between the stations 18, 20, preferably before the pH reduction station 26—see press station 19' illustrated in Figure 2.

The bleached mechanical pulp produced according to the sequence illustrated in Figure 1 has high brightness; brightness on the order of about 83% ISO units, or more, can be achieved. Almost as high a brightness can be achieved if a peroxide stage is utilized both before and after a hydrosulfite stage. This is shown schematically in Figure 5 wherein T stage 20' is between P₁ stage 12' and P₂ stage 18'. The following examples illustrate the enhanced brightness that can be achieved by the practice of the method of the invention.

Example 1 Prior art

A P-T sequence was practiced on unbleached CTMP pulp having 53.5% ISO, 160/110 (cold/hot) ml C.S.F. (millimeters, Canadian Standard Freeness) containing 0.4% DCM-extract (a measure of the amount of resin), 92 ppm manganese, and 9 ppm iron. For the peroxide stage, the pulp was pretreated with 0.2% DTPA (pentasodium salt of diethylenetriamine pentaacetic acid on bone dry (BD) pulp at 3% consistency and 60°C. The

temperature in both the P and T stages was 80°C, and the pulp consistency was 15% in both stages. The treatment time in the peroxide stage was 180 minutes using 3% sodium silicate and 0.2% MgSO₄ for buffering and stabilizing the peroxide liquid. In the T stage the reaction time was 30 minutes, using 1% hydrosulfite which was all consumed. The following results were obtained:

Peroxide stage

Charge H ₂ O ₂ , %	5.0
Consumption H ₂ O ₂ , %	3.3
Charge NaOH, %	3.5
End-pH	9.3
Brightness, % ISO	77.8

Hydrosulfite stage

End pH	5.5
Brightness, % ISO	78.7

Example 2

Practicing the present invention utilizing the same pulp as described in Example 1, pretreated in the same way, with the same buffering and stabilizing agents, with the same temperatures (i.e. 80°C) and consistency (i.e. 15% in all stages), and with a reaction time of 180 minutes in each peroxide stage and 30 min. in the hydrosulfite stage (1%, which was all consumed), the following results were obtained:

Peroxide stage 1

Charge H ₂ O ₂ , %	3.0
Consumption H ₂ O ₂ , %	2.3
Charge NaOH, %	3.0
End-pH	9.5
Brightness, % ISO	75.4

Peroxide stage 2

Charge H ₂ O ₂ , %	5.0
Consumption H ₂ O ₂ , %	2.9
Charge NaOH, %	3.5
End-pH	10.5
Brightness, % ISO	80.7

Hydrosulfite stage

End pH	6.1
Brightness, % ISO	83.5

Between the second peroxide stage and the hydrosulfite stage the pulp was pressed and the pH was reduced by the addition of SO₂ water, the reduction practiced so that the pH was about 5.5—6.0 just prior to the hydrosulfite stage, and the pulp was diluted as necessary in order to maintain the consistency throughout the actual bleaching stages at about 15%.

Example 3

In this example the hydrosulfite stage was between two peroxide stages (see Fig. 5). The pulp, and general conditions, were the same as for Example 2, with the following results:

Peroxide stage 1

Charge H ₂ O ₂ , %	3.0
Consumption H ₂ O ₂ , %	2.5
Charge NaOH, %	3.0
End-pH	9.3
Brightness, % ISO	75.4

Hydrosulfite stage

End pH	6.3
Brightness, % ISO	77.7

Peroxide stage 2

Charge H ₂ O ₂ , %	5.0
Consumption H ₂ O ₂ , %	3.2
Charge NaOH, %	3.5
End-pH	10.2
Brightness, % ISO	82.6

The final pulp brightness of almost 83 ISO (82.6) was surprisingly high, although not quite as high as the one for the sequence in Example 2.

It will thus be seen that according to the present invention a mechanical pulp having high brightness is produced, with a synergistic effect being achieved in pulp brightness by utilizing multiperoxide stages prior to a hydrosulfite stage.

Claims

1. A method of obtaining high brightness lignocellulose pulp comprising the steps of subjecting a mechanical lignocellulose pulp to peroxide bleaching and a hydrosulfite bleaching sequence (20), characterized by subjecting the mechanical pulp to a first peroxide bleaching (21), and then to a second peroxide bleaching (18), and then to the hydrosulfite bleaching sequence (20), to produce a bleached mechanical pulp.

2. A method as claimed in claim 1, characterized in that the consistency of the pulp during the practice of each of the steps is within the range of 8—30%.

3. A method as claimed in claim 2, characterized in that the consistency of the pulp during the hydrosulfite bleaching sequence is within the range of 8—15%.

4. A method as claimed in claim 3, comprising the further step of prior to the hydrosulfite bleaching sequence, reducing the pH of the mechanical pulp to between 5.5—6.0 (26).

5. A method as claimed in claim 1, comprising the further step, between the peroxide bleaching steps, of pressing the mechanical pulp (19).

6. A method as claimed in claim 1, wherein the first and second peroxide bleaching steps and the hydrosulfite bleaching sequence are the only

bleaching steps practiced in the treatment of the mechanical pulp.

7. A high brightness mechanical lignocellulose pulp, characterized in that it is produced by the sequential steps of: (a) subjecting a mechanical lignocellulose pulp to a first peroxide bleaching sequence (12); then (b) subjecting the mechanical pulp to a second peroxide bleaching sequence (18); and then (c) subjecting the mechanical pulp to a hydrosulfite bleaching sequence (20).

8. A high brightness pulp as claimed in claim 7, characterized in that it has a brightness of 83% ISO.

Patentansprüche

1. Verfahren zum Erhalten einer Lignozellulosepulpe von hohem Weißgrad, wobei eine mechanische Lignozellulosepulpe einem Peroxidbleichvorgang und einem Hydrosulfitbleichvorgang (20) unterworfen wird, dadurch gekennzeichnet, daß die mechanische Pulpe einem ersten Peroxidbleichvorgang (21), darauf einem zweiten Peroxidbleichvorgang (18) und danach dem Hydrosulfitbleichvorgang (20) unterworfen wird, um eine gebleichte mechanische Pulpe herzustellen.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Konsistenz der Pulpe während der Ausführung eines jeden der Schritte im Bereich von 8—30% liegt.

3. Verfahren nach Anspruch 2, dadurch gekennzeichnet, daß die Konsistenz der Pulpe während des Hydrosulfitbleichvorganges im Bereich von 8—15% liegt.

4. Verfahren nach Anspruch 3, mit dem weiteren Schritt, daß vor dem Hydrosulfitbleichvorgang der pH-Wert der mechanischen Pulpe auf 5,5—6,0 herabgesetzt wird (26).

5. Verfahren nach Anspruch 1, bei welchem in einem weiteren Schritt zwischen den Peroxidbleichschritten die mechanische Pulpe gepreßt wird (19).

6. Verfahren nach Anspruch 1, bei welchem der erste und der zweite Peroxidbleichschritt und der Hydrosulfitbleichvorgang die einzigen zur Behandlung der mechanischen Pulpe ausgeübten Bleichschritte sind.

7. Mechanische Lignozellulosepulpe von hohem Weißgrad, dadurch gekennzeichnet, daß sie durch die nachstehend angeführten aufeinanderfolgenden Schritte hergestellt ist: (a) eine mechanische Lignozellulosepulpe wird einem ersten Peroxidbleichvorgang (12) unterworfen; dann (b) wird die mechanische Pulpe einem zweiten Peroxidbleichvorgang (18) unterworfen; und danach (c) wird die mechanische Pulpe einem Hydrosulfitbleichvorgang (20) unterworfen.

8. Pulpe von hohem Weißgrad nach Anspruch 7, dadurch gekennzeichnet, daß sie einen Weißgrad von 83% ISO hat.

Revendications

1. Procédé permettant d'obtenir une pâte de

lignocellulose de haute brillance, qui consiste à soumettre une pâte mécanique de lignocellulose à un blanchiment au peroxyde et à un blanchiment à l'hydrosulfite (20), caractérisé en ce qu'on soumet la pâte mécanique à un premier blanchiment au peroxyde (21) et ensuite, à un second blanchiment au peroxyde (18) et ensuite à un blanchiment à l'hydrosulfite (20) pour obtenir une pâte mécanique blanchie.

2. Procédé selon la revendication 1, caractérisé en ce que la consistance de la pâte pendant la mise en oeuvre de chacune des étapes est comprise entre 8 et 30%.

3. Procédé selon la revendication 2, caractérisé en ce que la consistance de la pâte pendant la séquence de blanchiment à l'hydrosulfite est comprise entre 8 et 15%.

4. Procédé selon la revendication 3, qui comporte une étape supplémentaire qui précède la séquence de blanchiment à l'hydrosulfite, consistant à réduire le pH de la pâte mécanique à une valeur entre 5,5 et 6,0 (26).

5. Procédé selon la revendication 1, comportant l'étape supplémentaire entre les étapes de blanchiment au peroxyde, consistant à presser la pâte mécanique (19).

6. Procédé selon la revendication 1, dans lequel la première et la seconde étapes de blanchiment au peroxyde et le blanchiment à l'hydrosulfite sont les seules étapes de blanchiment qu'on utilise dans le traitement de la pâte mécanique.

7. Pâte mécanique de lignocellulose à haute brillance, caractérisée en ce qu'on la produit par les étapes successives, consistant à: (a) soumettre une pâte mécanique de lignocellulose à une première séquence de blanchiment au peroxyde (12); ensuite (b) soumettre la pâte mécanique à une seconde séquence de blanchiment au peroxyde (18); et (c) soumettre la pâte mécanique à une séquence de blanchiment à l'hydrosulfite (20).

8. Pâte de haute brillance selon la revendication 7, caractérisée en ce que sa brillance est de 83% ISO.

25

30

35

40

45

50

55

60

65

5

