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(54) Process for bleaching of lignocellulose-containing pulp

Verfahren zum Bleichen von Lignocellulose-enthaltendem Zellstoff

Procédé pour le blanchiment de pâte à papier contenant de la lignocellulose

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EP 0 679 760 B1

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Description

5 The present invention relates to a process for delignification and bleaching of chemically digested lignocellulose-containing pulp, wherein the pulp is acid treated at a pH of between about 1 and about 6, whereupon a water-soluble mixture of a magnesium compound and a calcium compound is added at a pH of between about 1 and about 7 before the pulp is treated with a chlorine-free bleaching agent comprising hydrogen peroxide at a pH of from about 8 up to about 12. The initial acidic treatment removes the trace metals of the pulp, whereas the subsequent addition of alkaline earth metal ions in aqueous solution returns the ions to the positions in the pulp where they have a particularly beneficial effect on the preservation of the cellulose chains and, consequently, on the viscosity, as well as on the consumption of bleaching agent in the subsequent bleaching step. After the treatment according to the invention, the pulp may be finally bleached to the desired brightness, suitably with a chlorine-free bleaching agent, such a ozone, to completely avoid formation and discharge of AOX.

Background

15 In the production of chemical pulp of high brightness, wood chips are first cooked to separate the cellulose fibres. Part of the lignin holding the fibres together is thus degraded and modified, such that it can be removed by subsequent washing. However, in order to obtain sufficient brightness, more lignin has to be removed, together with brightness-impairing (chromophoric) groups. This is frequently effected by delignification with oxygen, followed by bleaching in several stages.

20 For environmental reasons, it has become increasingly common to treat chemical pulp with chlorine-free bleaching agents already in the first bleaching steps. The big advantage is the drastic reduction in the discharges of chlorinated organic substances detrimental to the environment, owing to the combined effect of a smaller amount of chlorine-containing bleaching agents and lower content of lignin, which is the organic substance primarily reacting with the chlorine.

25 It is known to use chlorine-free bleaching agents, such as hydrogen peroxide, peracetic acid or ozone, already in the prebleaching. However, the delignification and consumption of the bleaching agent become less effective than with chlorine-containing bleaching agents, unless the pulp is pretreated. Thus, a hydrogen peroxide treatment in an alkaline environment is disturbed by the presence in the pulp of ions of certain metals, such as Mn, Cu and Fe. These metal ions cause degradation of hydrogen peroxide, thereby reducing the efficiency of the peroxide treatment and increasing the consumption of peroxide. According to CA-A-1,206,704, this can be counteracted by pretreating the pulp with an acid, such as sulphuric acid or nitric acid, whereby the concentration of all types of metal ions is reduced. However, by this treatment also metal ions, for example Mg, which are advantageous to the peroxide treatment disappear, which ions stabilize the peroxide and increase the selectivity of the peroxide.

30 CA-A-575,636 discloses the addition of magnesium sulphate to stabilize alkaline peroxide solutions. However, the addition is made directly to the bleaching liquor and in alkaline environment insoluble magnesium hydroxide precipitates. EP-A-0 148 712 teaches to bleach chemical cellulose pulp with hydrogen peroxide in one step at a pH of 11 to 11,5. The peroxide bleach solution comprises a salt of magnesium and a salt of calcium which are kept in solution by means of a complexing agent. Furthermore, US-A-4,222,819 discloses the addition of magnesium ions to acidic peroxide solutions, but also in this case the addition is made directly to the bleaching liquor. None of the related methods makes possible diffusion of the magnesium ions into the pulp to such an extent, that a pulp of high brightness and strength can be obtained.

The invention

45 The invention provides a process in which lignocellulose-containing pulp is treated under the conditions disclosed in the claims, whereby the metal ions harmful to the subsequent bleaching are effectively removed and the profile of alkaline earth metals is restored before the pulp is bleached in a chlorine-free bleaching step.

50 The invention relates to a process for bleaching of chemically digested lignocellulose-containing pulp, wherein the pulp is acid treated at a pH in the range from about 1 up to about 6, whereupon the pulp is washed and a mixture containing a magnesium compound and a calcium compound is added at a pH in the range from about 1 up to about 7 and in an amount of from about 0.01 kg, suitably 0.5 kg, up to about 10 kg/ton of dry pulp, calculated as magnesium and calcium, and that the pulp subsequently is treated with a chlorine-free bleaching agent comprising hydrogen peroxide at a pH of from about 8 up to about 12.

55 Acid treatment is an effective process to eliminate metal ions from lignocellulose-containing pulps. At the same time it is known, that ions of magnesium and calcium, especially when in their original positions in the pulp, have a positive influence on the selectivity of the delignification as well as on the stability and consumption of chlorine-free bleaching agents, such as peroxides, ozone and oxygen. The present process presents an economic solution to the

problem of creating a suitable trace-metal profile for the subsequent chlorine-free bleaching, in that non-desirable metal ions are eliminated while supplied ions of magnesium and calcium essentially recover the positions in the vicinity of the cellulose chains previously occupied by ions of alkaline earth metals. This is achieved by adding a mixture of a magnesium compound and a calcium compound at such a pH and such a temperature that the compound is dissolved in water, thus enabling the diffusion required to obtain the intended effect. Furthermore, an advantage of the present process is that the pH adjustment between the treatment with acid and addition of alkaline earth metal ions becomes very limited or may be left out altogether, which is advantageous to process technique and economy.

Chlorine-free bleaching agents include inorganic peroxide compounds, such as hydrogen peroxide and sodium peroxide, organic peroxide compounds, such as peracetic acid, as well as ozone, oxygen and sodium dithionite. Suitably, hydrogen peroxide (P), oxygen (O) and ozone (Z) are used in an optional sequence or mixture. Preferably, use is made of hydrogen peroxide or mixtures of hydrogen peroxide and oxygen (PO). The sequence P-Z or (PO)-Z are especially preferred.

In the treatment with a chlorine-free bleaching agent in an alkaline environment, pH is suitably adjusted by adding to the pulp an alkali or an alkali-containing liquid, such as sodium carbonate, sodium hydrogen carbonate, sodium hydroxide, oxidized white liquor or magnesium hydroxide slurry. Suitably, the magnesium hydroxide slurry is taken from the chemical handling system in the production of sulphite pulp with magnesium as base, i.e. magnesite pulp.

The acid treatment suitably is carried out with an acid. The acids used are inorganic acids, suitably sulphuric acid, nitric acid, hydrochloric acid or residual acid from a chlorine dioxide reactor, either separately or in an optional mixture. Preferably, sulphuric acid is employed.

Use is suitably made of magnesium-containing compounds, such as magnesium sulphate or magnesium chloride, and calcium-containing compounds, such as calcium chloride or calcium oxide. The combination of temperature and pH at the addition of the mixture of a magnesium compound and a calcium compound is always so chosen that the compound is in aqueous solution when contacted with the pulp.

In the process according to the invention, the acid treatment is carried out at a pH of from about 1 up to about 6, suitably from 1.5 up to 5, preferably from 2 up to 4. It is especially preferred that the acid treatment is carried out at a pH of from 2 up to 3. Magnesium is one of the alkaline earth metals in the compounds containing an alkaline earth metal. The addition is made at a pH in the range from about 1 up to about 7, suitably in the range from 2 up to 6, preferably in the range from 2 up to 4. It is especially preferred that the addition of magnesium is made at a pH of from 2 up to 3. When the chlorine-free bleaching agent is hydrogen peroxide, the pulp is treated at a pH of from about 8 up to about 12, preferably at a pH of from 10 up to 12. Treatment with the other chlorine-free bleaching agents mentioned above, is carried out within the same pH range.

The treatment according to the invention is carried out with a washing step between the acid treatment and addition of alkaline earth metal ions, such that the trace metals that are harmful to the treatment with a chlorine-free bleaching agent are removed from the pulp suspension.

The realization of the acid treatment, a mixture of a magnesium compound and a calcium compound and a chlorine-free bleaching agent, can be carried out at an optional position in the bleaching sequence, e.g. immediately after digestion of the pulp or after an oxygen step. The process according to the invention is preferably applied to pulp that has been delignified in an oxygen step prior to the treatment.

It is also within the scope of the invention, that the pulp in the acid treatment also can be subjected to bleaching and/or delignifying treatment. Bleaching and/or delignifying chemicals active within the pH range suitable in the acid treatment, are e.g. chlorine dioxide, ozone, peracetic acid and/or an acid peroxide-containing compound. Suitably, a combination of acid treatment and bleaching and/or delignifying treatment takes place in an ozone step.

Lignocellulose-containing pulps relate to chemical pulps of softwood and/or hardwood digested according to the sulphite, sulphate, soda or organosolv process, or modifications and/or combinations thereof. Use is suitably made of softwood and/or hardwood digested according to the sulphate process, preferably sulphate pulp of hardwood.

The treatment according to the invention can be applied to lignocellulose-containing pulps having an initial kappa number within the range from about 5 up to about 40, suitably 7 up to 32, preferably from 10 up to 20. Here, the kappa number is determined according to the standard method SCAN-C 1:77.

In the process according to the invention, the acid treatment is carried out at a temperature of from about 10 up to about 95°C, suitably from 20 up to 80°C and preferably from 40 up to 80°C, and for a period of time of from about 1 up to about 120 min, suitably from 10 up to 120 min and preferably from 20 up to 40 min. The mixture of a magnesium compound and a calcium compound is added at a temperature of from about 10 up to about 95°C, preferably from 40 up to 80°C, and for a period of time of from about 1 up to about 180 min, preferably from 20 up to 180 min and preferably from 30 up to 120 min. When the chlorine-free bleaching agent is hydrogen peroxide, the pulp is treated at a temperature of from about 30 up to about 100°C, preferably from 60 up to 90°C, and for a period of time of from about 30 up to about 300 min, suitably from 60 up to 240 min. In the acid treatment and in the addition of a mixture of magnesium and calcium ions, the pulp concentration may be from about 3 up to about 35% by weight, preferably from 3 up to 15% by weight. When the chlorine-free bleaching agent is hydrogen peroxide, the pulp concentration may be from about 3

up to about 50% by weight, suitably from 3 up to 35% by weight and preferably from 10 up to 25% by weight. Treatment with the other chlorine-free bleaching agents mentioned above, is carried out within the normal ranges of temperature, time and pulp concentration for each bleaching agent, which are well-known to the person skilled in the art.

The amount of mixture containing a magnesium compound and a calcium compound charged, lies in the range from about 0.5 up to about 10 kg/ton of dry pulp, suitably in the range from 0.5 up to 5 kg/ton of dry pulp, and preferably in the range from 2 up to 4 kg/ton of dry pulp, calculated as magnesium and calcium.

In preferred embodiments employing hydrogen peroxide as the chlorine-free bleaching agent, the amount of hydrogen peroxide, lies in the range from about 2 up to about 50 kg/ton of dry pulp, calculated as 100% hydrogen peroxide. The upper limit is not critical, but has been set for reasons of economy. The amount of hydrogen peroxide suitably lies in the range from 3 up to 30 kg/ton of dry pulp and preferably from 4 up to 20 kg/ton of dry pulp, calculated as 100% hydrogen peroxide.

After the acid treatment, washing and treatment with a mixture of a magnesium compound and a calcium compound, and a chlorine-free bleaching agent, the pulp can be used for direct production of paper with a lower demand of brightness. Alternatively, the pulp may be finally bleached to the desired higher brightness, by treatment in one or more steps. Suitably, the final bleaching is also carried out with chlorine-free bleaching agents of the type mentioned above, optionally with intermediate alkaline extraction steps, which may be reinforced with peroxide and/or oxygen. In this way, the formation and discharge of AOX is completely eliminated. Suitably, the final bleaching is carried out with ozone in one or more steps. By the treatment according to the invention, the lignin content has been reduced to a sufficiently low level before any chlorine-containing bleaching agents are used. Therefore, chlorine dioxide and/or hypochlorite may well be used in one or more final bleaching steps without causing formation of large amounts of AOX.

Moreover, use of the process according to the invention means that the brightness and kappa number of the resulting pulp is higher and lower, respectively, than with the processes in which a mixture of a magnesium compound and a calcium compound is not added at all or is added at a higher pH. In a process for bleaching chemical pulps, the aim is a high brightness as well as a low kappa number, the latter meaning a low content of undissolved lignin. At the same time, the consumption of the chlorine-free bleaching agent should be as low as possible meaning lower treatment costs. In the process according to the invention, these objects are met. Furthermore, the strength of the pulp, measured as viscosity, is sufficient, which means that the pulp contains cellulose chains which are long enough to give a strong product.

The invention and its advantages are illustrated in more detail by the example below which, however, is not part of the invention, but is only intended to illustrate the effect of magnesium at various pH values. The percentages and parts stated in the description, claims and examples, refer to percent by weight and parts by weight, respectively, unless anything else is stated.

Example

Sulphate pulp of softwood having a kappa number of 17, a brightness of 35% ISO and a viscosity of 970 dm³/kg was treated with sulphuric acid at a pH of 2.0. The pulp was treated at a temperature of 60°C for 30 min, the pulp concentration being 10% by weight. After washing the pulp with water, magnesium was added in the form of an aqueous solution containing MgSO₄, to give a concentration of magnesium in the pulp of at least 500 ppm. In the tests, the pH at the time of the addition was varied between 2.3 and 11.5 by addition of sulphuric acid. Then, the pulp was bleached with hydrogen peroxide at a temperature of 90°C, the residence time and pulp concentration being 180 min and 15% by weight, respectively.

The final pH was 11.5, and the addition of hydrogen peroxide was 15 kg/ton of dry pulp, calculated as 100% hydrogen peroxide. For comparative purposes, magnesium was added directly to the hydrogen peroxide step under the conditions stated above, in accordance with the prior art. To provide a further comparison, the pulp was also treated with only sulphuric acid and hydrogen peroxide under the conditions stated above. The kappa number, viscosity and brightness of the pulp were determined according to SCAN Standard Methods, and the consumption of hydrogen peroxide was determined by iodometric titration. The test results appear from the Table below.

TABLE I

pH at the addition of Mg	Kappa number step 2	Viscosity step 2 (dm ³ /kg)	Brightness step 2 (% ISO)	Residual H ₂ O step 2 (kg/ton)
2.3	9.1	903	61.0	1.5
4.7	9.2	910	60.0	1.0
9.5	9.8	930	56.1	0.9
11.5	10.0	940	52.2	0.2

TABLE I (continued)

pH at the addition of Mg	Kappa number step 2	Viscosity step 2 (dm ³ /kg)	Brightness step 2 (% ISO)	Residual H ₂ O step 2 (kg/ton)
---- *	9.8	890	54.1	0.5
2.3 **	9.9	875	48.2	0.0

* Magnesium added directly to the alkaline hydrogen peroxide step.

** No magnesium added.

As is apparent from the Table, the treatment with MgSO₄ at a pH in the range from about 2 up to about 6 is essential to give maximum increase in brightness and maximum reduction of the kappa number, as well as minimum decrease in viscosity and minimum consumption of hydrogen peroxide. Furthermore, the importance of the magnesium ions for the increase in brightness appears from the comparison at a pH of 2.3, where, in the final test, the peroxide treatment was preceded only by acidic treatment.

Claims

1. A process for delignification and bleaching of chemically digested lignocellulose-containing pulp, during which the pulp is acid treated at a pH in the range from about 1 up to about 6 and the pulp is washed after the acid treatment, **characterised** in that thereafter a water-soluble mixture of a magnesium compound and a calcium compound is added at a pH in the range from about 1 up to about 7 and in an amount of from about 0.5 up to about 10 kg/ton of dry pulp, calculated as magnesium and calcium, and that subsequently the pulp is delignified and bleached with a chlorine-free bleaching agent comprising hydrogen peroxide at a pH of from about 8 up to about 12.
2. A process according to claim 1, **characterised** in that the water-soluble mixture of a magnesium compound and a calcium compound is added in an amount of from about 2 up to about 10 kg/ton of dry pulp, calculated as magnesium and calcium.
3. A process according to claim 1, **characterised** in that the chemically digested pulp is a sulphate pulp.
4. A process according to claim 1, **characterised** in that the water-soluble mixture of a magnesium compound and a calcium compound is added at a pH of from 2 up to 6.
5. A process according to claim 1, **characterised** in that the calcium compound is calcium chloride or calcium oxide.
6. A process according to claim 1 or 5, **characterised** in that magnesium compound is magnesium sulphate or magnesium chloride.
7. A process according to claim 1, **characterised** in that the chlorine-free bleaching agent consists of oxygen and hydrogen peroxide.
8. A process according to claim 7, **characterised** in that the pulp, after the treatment, is finally bleached with ozone in one or more steps.
9. A process according to claims 1-8, **characterised** in that the acid treatment is carried out at a temperature of from about 10 up to about 95°C for about 1 up to about 120 min, that the mixture of a magnesium compound and a calcium compound is added at a temperature of from about 10 up to about 95°C for about 1 up to about 180 min and in an amount of from 0.5 up to 5 kg/ton of dry pulp, calculated as magnesium compound and calcium compound, the treated pulp having a concentration of from about 3 up to about 35% by weight, and that the pulp is bleached and delignified with hydrogen peroxide at a pH of from about 8 up to about 12.

Patentansprüche

1. Verfahren zum Entholzen und Bleichen von chemisch aufgeschlossenem lignocellulosehaltigem Zellstoff, in dem der Zellstoff bei einem pH-Wert im Bereich von etwa 1 bis etwa 6 mit Säure behandelt wird und der Zellstoff nach

EP 0 679 760 B1

der Säurebehandlung gewaschen wird, dadurch gekennzeichnet, daß danach ein wasserlösliches Gemisch einer Magnesiumverbindung und einer Calciumverbindung bei einem pH-Wert im Bereich von etwa 1 bis etwa 7 und in einer Menge von etwa 0.5 bis etwa 10 kg/Tonne des trockenen Zellstoffs, berechnet als Magnesium und Calcium, zugegeben wird, und daß der Zellstoff anschließend mit einem chlorfreien Bleichmittel, umfassend Wasserstoffperoxid, bei einem pH-Wert von etwa 8 bis etwa 12 entholzt und gebleicht wird.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das wasserlösliche Gemisch einer Magnesiumverbindung und einer Calciumverbindung in einer Menge von etwa 2 bis etwa 10 kg/Tonne des trockenen Zellstoffs, berechnet als Magnesium und Calcium, zugegeben wird.

3. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der chemisch aufgeschlossene Zellstoff Sulfatzellstoff ist.

4. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das wasserlösliche Gemisch einer Magnesiumverbindung und einer Calciumverbindung bei einem pH-Wert von 2 bis 6 zugegeben wird.

5. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Calciumverbindung Calciumchlorid oder Calciumoxid ist.

6. Verfahren nach Anspruch 1 oder 5, dadurch gekennzeichnet, daß die Magnesiumverbindung Magnesiumsulfat oder Magnesiumchlorid ist.

7. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das chlorfreie Bleichmittel aus Sauerstoff und Wasserstoffperoxid besteht.

8. Verfahren nach Anspruch 7, dadurch gekennzeichnet, daß der Zellstoff nach der Behandlung schließlich in einem oder mehreren Schritten mit Ozon gebleicht wird.

9. Verfahren nach den Ansprüchen 1-8, dadurch gekennzeichnet, daß die Säurebehandlung bei einer Temperatur von etwa 10 bis etwa 95°C während etwa 1 bis etwa 120 Min. durchgeführt wird, daß das Gemisch einer Magnesiumverbindung und einer Calciumverbindung bei einer Temperatur von etwa 10 bis etwa 95°C während etwa 1 bis etwa 180 Min. und in einer Menge von 0.5 bis 5 kg/Tonne des trockenen Zellstoffs, berechnet als Magnesiumverbindung und Calciumverbindung, zugegeben wird, wobei der behandelte Zellstoff eine Konzentration von etwa 3 bis etwa 35 Gew.-% hat, und daß der Zellstoff bei einem pH-Wert von etwa 8 bis etwa 12 mit Wasserstoffperoxid gebleicht und entholzt wird.

Revendications

1. Procédé de délignification et de blanchiment de pâte à papier contenant de la lignocellulose, qui a été soumise à une digestion par voie chimique, procédé dans lequel on traite la pâte avec un acide, à un pH compris dans l'intervalle allant d'environ 1 à environ 6, et on lave la pâte après le traitement par un acide, **caractérisé** en ce que l'on ajoute ensuite un mélange soluble dans l'eau d'un dérivé du magnésium et d'un dérivé du calcium, à un pH compris dans l'intervalle allant d'environ 1 à environ 7 et en une quantité comprise entre environ 0,5 et environ 10 kg par tonne de pâte sèche, ladite quantité étant calculée en magnésium et en calcium, et en ce que la pâte est ensuite délignifiée et blanchie au moyen d'un agent de blanchiment exempt de chlore, renfermant du peroxyde d'hydrogène, à un pH compris dans l'intervalle allant d'environ 8 à environ 12.

2. Procédé selon la revendication 1, **caractérisé** en ce que le mélange soluble dans l'eau d'un dérivé du magnésium et d'un dérivé du calcium est ajouté en une quantité comprise entre environ 2 et environ 10 kg par tonne de pâte sèche, ladite quantité étant calculée en magnésium et en calcium.

3. Procédé selon la revendication 1, **caractérisé** en ce que la pâte qui a été soumise à une digestion par voie chimique, est une pâte au sulfate.

4. Procédé selon la revendication 1, **caractérisé** en ce que le mélange soluble dans l'eau d'un dérivé du magnésium et d'un dérivé du calcium est ajouté à un pH compris entre 2 et 6.

EP 0 679 760 B1

5. Procédé selon la revendication 1, **caractérisé** en ce que le dérivé du calcium est le chlorure de calcium ou l'oxyde de calcium.
- 5 6. Procédé selon la revendication 1 ou 5, **caractérisé** en ce que le dérivé du magnésium est le sulfate de magnésium ou le chlorure de magnésium.
7. Procédé selon la revendication 1, **caractérisé** en ce que l'agent de blanchiment exempt de chlore est constitué d'oxygène et de peroxyde d'hydrogène.
- 10 8. Procédé selon la revendication 7, **caractérisé** en ce que, après le traitement, la pâte est soumise à un blanchiment final par l'ozone, en une ou plusieurs étapes.
- 15 9. Procédé selon l'une quelconque des revendications 1 à 8, **caractérisé** en ce que l'on réalise le traitement par un acide à une température comprise dans l'intervalle allant d'environ 10 à environ 95 °C, pendant une durée allant d'environ 1 à environ 120 minutes, en ce que le mélange d'un dérivé du magnésium et d'un dérivé du calcium est ajouté à une température comprise dans l'intervalle allant d'environ 10 à environ 95 °C, en un laps de temps allant d'environ 1 à environ 180 minutes et en une quantité comprise dans l'intervalle allant de 0,5 à 5 kg par tonne de pâte sèche, ladite quantité étant calculée en magnésium et en calcium et la pâte traitée ayant une concentration d'environ 3 à environ 35 % en poids, et en ce que la pâte est blanchie et délignifiée au moyen de peroxyde d'hydrogène, à un pH compris dans l'intervalle allant d'environ 8 à environ 12.
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