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(54) **METHOD AND ARRANGEMENT FOR TREATING FILTRATE AFTER OXYGEN DELIGNIFICATION OF CHEMICAL PULP COOKED TO A HIGH KAPPA NUMBER**

VERFAHREN UND ANORDNUNG ZUR BEHANDLUNG EINES FILTRATS NACH DER SAUERSTOFFDELIGNIFIZIERUNG EINES BEI HOHER KAPPA-ZAHL ERHITZTEN CHEMISCHEN ZELLSTOFFS

PROCÉDÉ ET DISPOSITIF DE TRAITEMENT D'UN FILTRAT APRÈS UNE DÉLIGNIFICATION PAR L'OXYGÈNE D'UNE PÂTE CHIMIQUE CUITE À UN NOMBRE KAPPA ÉLEVÉ

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Description

[0001] The present invention relates to a method and an arrangement for treating chemical pulp when the pulp has been cooked to a high kappa.

[0002] The kappa number indicates the residual lignin content and it is used at all mills as a target value for cooking. A typical kappa number for pulp to be bleached is at present 14-20 for hard wood and 25-35 for soft wood. If the pulp is not bleached, the kappa number after cooking is clearly higher, typically 40-100. Soft wood pulp to be bleached is typically treated in an oxygen stage for decreasing the kappa number to 10-20.

[0003] In connection with the invention, a high kappa pulp refers to kappa numbers exceeding 50. High kappa pulps (high yield pulps) are usually used in products, from which high strength properties and rigidity are required, but not brightness or printing properties. For this reason, soft wood is a typical raw material for high yield pulps due to its long fibers. Soft wood has a high lignin content, for which it requires a relatively long chemical treatment. A combination of strength properties and rigidity is typical for soft wood pulp. For high yield pulps, the lignin content remains so high that in prior art technique the pulps can not be defibrated without mechanical treatment, but in this case the final product does not require printing properties or brightness.

[0004] High yield pulps are typically produced in a sulfate process. The yield range of high yield pulps varies in the range of 50-70%. It is common knowledge that this kind of pulp is not produced using fiber line equipment designed for pulp with a normal kappa number, but additional technique is required. The most important difference between the fiber lines is that high kappa pulp after cooking is in such a strong chip form that the defibering thereof requires special defibering equipment, such as a blow line refiner (In-Line refiner), or alternatively hot refining. The arrangements are of similar type. In the first-mentioned defibering arrangement, cooked pulp in the form of chips is discharged from the digester into a refiner located in the blow line upstream of washing stages. The aim of the defibration of cooked pulp is to separate the fibers from each other, since after cooking the fibers are still in the chip matrix. This defibration allows producing pulp to a high kappa and to high yield values. Even a slight defibration is mechanical treatment of the pulp, the effects of which in alkaline conditions decrease the quality of the pulp.

[0005] High kappa pulp is produced to so-called brown stock. Thus, there is no aim to bleach this kind of pulp. The production line for this pulp comprises washing and screening of the pulp. If any special property requirements are set for the pulp quality, the line may comprise an oxygen stage for adjusting the properties of the pulp to a desired level.

[0006] Dissolving of carbohydrates (cellulose and hemicelluloses) during cooking causes a remarkable increase in the raw material costs, since cabrohydrate losses in both the beginning and at the end of cooking are big. One way of decreasing the decomposing of carbohydrates is to end the cook at a higher kappa number. Typically, the yield is increased, if the cook is ended at a high kappa number and the pulp is bleached with chlorine dioxide without an oxygen stage. Still, the costs of bleaching and increasing effluent discharges exceed the allowed limits. Keeping the effluent discharges at the BAT (Best available techniques) mill level requires the use of an oxygen stage, whilst so that the carbohydrate yield is the highest possible. Vigorous development of oxygen delignification has provided an efficient method of decreasing the kappa of pulp to be bleached without substantial losses in yield. The present trend has been to stop reaching cooking kappa numbers of remarkably low level.

[0007] Important in sulfate cooking is selectivity, i.e. when lignin is dissolved, a least possible amount of carbohydrates (cellulose and hemicelluloses) is split and dissolved. A problem is caused by chemical reactions, due to which a remarkable amount of hemicellulose is dissolved at the finalizing stage of the cook. Thus, at the final stage dissolving of carbohydrates also causes a decrease in the strength of chemical pulp. In the case of softwood pulp, the strength of chemical pulp is one of the most essential quality indicators. In addition to pulp properties, also process yield is an important cost factor. The aim in the past years has been to find such pulping methods, by means of which the pulp yield from wood would be the highest possible. It has meant either increasing the selectivity of the cook or cooking only to such a kappa number that the use of more selective delignification methods provides remarkable yield benefit and thus economical importance The differences between sulfate cooking methods in a final kappa range (for softwood 25-40) are so small that no substantial differences in terms of selectivity are obtained by means of them. In practice, a feasible method for increasing the yield is to finish the cook at a clearly higher kappa number level, where the carbohydrate yield is relatively higher than in cooks of pulp to be bleached and to continue the delignification with oxygen, which is known to be a more selective way of delignifying pulp compared to cooking.

[0008] Due to alkaline conditions, oxygen delignification is a direct continuation to lignin-removal taking place during cooking. In the industrial cooking processes the chemical pulp is not cooked to significantly below kappa number 25, since the yield loss would be essential. On the other hand, there is a desire to optimize the cook to a highest possible kappa number in order to maintain the reject content under control and the pulp bleachable. Between the cook and the final bleaching, oxygen delignification is a more selective and gentler process. It disintegrates and oxidizes lignin to a form that is dissoluble in alkali, destroys colored compounds present in lignin and removes impurities (resin) from the pulp.

[0009] In view of quality, defibrating the pulp after discharge from the digester in a blow line defibrator is never the so-called optimal solution, but defibrating the pulp chemically without remarkable mechanical loading preserves the fiber

properties best.

[0010] The latest cooking studies have revealed that cooking can produce high kappa pulp that is defiberazed to such an extent that this chip-form pulp can be pumped and treated in a fiber line even at a kappa number of 70-90 (Jiajun Wang, Master's Thesis, Aalto Yliopisto, School of Science and Technology 2010; WO Patent Application PCT/US10/57417). Also in this stage, the cooked pulp contains abundantly of analyzed reject, but the character of the reject is such that is does not limit the pumping of the pulp to washing and subsequent process stages.

[0011] FI patent application 20115277 presents a method of treating pulp that is produced by chemically cooking soft wood chips to a kappa number of 50-100, preferably 60-90, in which method

the cooked pulp is led to washing without treament in a mechanical defibrator between the digester and the washing, the pulp is washed,
the washed pulp is led into a first oxygen delignification stage where the pulp is treated in the presence of oxygen and alkali,
the oxygen delignified pulp is washed and after the washing it is treated in a subsequent stage with an electrophilic chemical, after which
the pulp is washed and treated in a second oxygen delignification stage with oxygen and alkali, and the thus treated pulp is washed and led to further treatment into a bleaching process.

[0012] In a publication "Oxygen Delignification of High-kappa Number Pine Kraft Pulp" by D. Danielewicz et al. in "Fibers & Textiles in Eastern Europe", vol. 14, no. 2, 2006, pages 89-93, a process is discussed in which pine kraft pulp having a kappa number of 68 is washed with distilled water and re-pulped. The pulp is then oxygen delignified to decrease the lignin content to 14 to 16 kappa number units and washed again with distilled water.

[0013] Oxygen stage in connection of the present patent application refers to such an alkaline stage that takes place pressurized within a pressure range of 1-20, preferably 8-12 bar (abs.) at the mixing point, at a pH range of 8.5-14 and wherein at least during part of the reaction time oxygen is present around the fibers. The oxygen stage can comprise one, two or even several steps, whereby each reaction step comprises chemical mixing and a reaction vessel or a reaction retention accomplished by means of a tube. In practice, treatment step here refers to addition and mixing of a chemical used in the oxygen stage and the subsequent retention in a portion of the tube or the reactor. Reaction retentions are, depending on the practicing method, from 0.1 minutes up to 120 minutes, so that the reaction retention depends on the desired type of reactor. In this connection, an oxygen stage is also known from a washing stage both upstream and downstream of the oxygen stage.

[0014] Most usually, oxygen and alkaline and possibly some inhibitor preventing fibers from getting damaged by metals are dosed into the oxygen stage, or metals entrained in the fibers are otherwise removed or treated to become inactive. Alkali is generally dosed in the amount of 1-60 kg/admt and oxygen 1-50 kg/admt. When the alkali dose is 0.8-1.4 x kappa unit and the oxygen dose 0.7-1.3 x kappa unit, it can be noted that the alkali dose of the oxygen stage may be up to 90 kg/adt and the oxygen dose 70 kg/adt, when the kappa reduction in the oxygen stage is 60 units. The alkali that is used is most often sodium hydroxide or oxidized white liquor, but in principle all alkaline compounds containing OH-ion are alkalies that could be used in the oxygen stage under some conditions. The make-up sodium hydroxide of the chemical cycle is often advantageously dosed in the oxygen stage. The oxygen is dosed in gaseous form, where the content of oxygen is most usually 75-100% of specific gravity. The temperature of the oxygen stage is 70 - 120 °C and most usually 80 - 105 °C. The temperature can be raised using some suitable steam having a pressure of 0.5-20 bar, and hot water either via washing or diluting. Steam can be used for heating either directly, mixed in the pulp, or indirectly.

[0015] In the oxygen delignification of high kappa pulp, especially in the first treatment, a large amount of lignin is released (the kappa number decreases by e.g. 30-70 units), which is to be taken into account in the process. When the amount of removed lignin is e.g. 40 kappa units, the amount of organic material being released from the pulp becomes high. With said kappa reduction, the proportion of e.g. lignin only is approximately 7% of the dry substance of the pulp, and thereto carbohydrate losses. Said amount of lignin raises the COD-amount of the filtrate of the oxygen stage by 60-120 kg/adt and the amount of dry substance in the same proportion. The oxygen stage is typically connected in accordance with the counter-current washing principle so that the function of the washing of the pulp arranged between the cooking and the oxygen stage is to displace the liquor entrained in the pulp coming from the washing, which liquor can be separated as a filtrate of the washing stage of the cooked pulp, with a filtrate obtained from the oxygen stage washing. Thus, the COD of the washing liquid of the cooked pulp will be in the range of 30-60 g/l and that of the pulp suspension in the range of 200-500 kg/adt at a consistency of 12%. Generally, filtrates from pulp washing are led counter-currently in the fiber line, but treating an oxygen stage filtrate having a high COD-value this way is not necessarily advantageous. Industrially known COD-levels are clearly lower than the above-mentioned in the studies that have been conducted for determining the behavior of filtrate generated from the oxygen stage, when it is led counter-currently to the same stage. The above presented levels are already so high that they hamper the operation of the oxygen stage

and cause consumption of chemical together with the circulating lignin. When outlining the oxygen stage for high kappa pulp, the amount of reaction products is to be taken into account.

[0016] Because the reaction products comprise, due to the selective character of the oxygen stage, lignin and a minor amount of carbohydrates, it can be stated that in the washing filtrate of the oxygen stage the proportion of lignin and carbohydrates is higher than in black liquor. Thus, the use thereof in washing of cooked pulp according to the counter-current principle is not advantageous without disturbing the oxygen stage process. Accordingly, a clear need exists to outline couplings of washing and oxygen stage for pulp cooked to a high kappa in a novel way, which is in many ways more practicable.

[0017] The present invention relates to a method of treating chemical pulp, in which method pulp is produced by cooking wood chips chemically to a kappa number of 50-120, preferably 60-100, the cooked pulp is washed, the washed pulp is led into an oxygen stage where the pulp is treated in the presence of oxygen and alkali for removing lignin, and the oxygen-treated pulp is washed, whereby lignin-containing washing filtrate is formed. The method is characterized in that

in the oxygen stage the kappa number of the pulp decreases by at least 30 units, and
the filtrate of the washing downstream of the oxygen stage is treated for separating lignin, and the treated filtrate is led as washing liquid into the washing apparatus for the cooked pulp. The E_{10}-value of the washing apparatus for cooked pulp is typically at least 8, preferably at least 10.

[0018] The so-called E10-value defines the washing efficiency so that washers of different types can be compared to each other. The E10-value is a numerical value defined for a washer or a combination of several washers, which reveals how many ideal mixings the washer or washer combination reaches. An ideal mixing, in turn, is understood as a situation when the washing liquid is mixed into the pulp being washed so efficiently that the concentrations of both the liquid remaining inside the pulp and the liquid being withdrawn therefrom are equal. Number 10 in the E_{10} value reveals the calculated consistency per cent, at which the pulp exits the washer. Dilution factor means the difference between used washing liquid and liquid exiting the washing apparatus with the pulp per a ton of pulp.

[0019] The invention also relates to an arrangement for treating pulp produced from wood chips in a cooking apparatus wherein the wood chips are cooked chemically to a kappa number of 50-120, preferably 60-100, said arrangement comprising at least

a cooking apparatus for cellulose fibrous material, washing devices for cooked pulp,
apparatuses downstream of washing of the cooked pulp for delignifying the pulp in an oxygen stage,
apparatuses for washing the pulp after the oxygen stage, and
filtrate lines for leading washing filtrates counter-currently to preceding washers as washing liquid. It is essential that a filtrate line downstream of the oxygen stage is provided with devices for separating lignin from the filtrate and that the filtrate line is connected to the washing devices for cooked pulp for using the filtrate as washing liquid after lignin separation.

[0020] The E10-value of the washing devices for cooked pulp is preferably at least 8, preferably at least 10. The washing devices for cooked pulp typically comprise more than one stage. The washing devices for cooked pulp typically comprise a so-called digester washing and at least one washing device between the digester and the oxygen treatment apparatuses.

[0021] The washing devices for cooked pulp can comprise as one stage a digester wash conducted in the lower part of the digester, such as a hi-heat type of digester displacement or final displacement taking place in batch cooking. The washing devices for cooked pulp comprise downstream thereof one or several washers, such as preferably a single drum washer, for instance a Drum Displacer® (DD) washer (Andritz Oy) or a diffuser washer. If the cooked pulp does not contain uncooked fraction in harmful amounts, such as knot, also a press can be used, under the precondition that the E_{10}-level of the overall washing devices for cooked pulp is over 8. The dilution factor of the washing devices is to be 0-5, preferably 1-3. Adequate efficiency of washing is important in order to ensure adequate removal of lignin released from the pulp during cooking. More than 95%, preferably more than 97% of lignin is removed in the washing. Simultaneously, also sulfur can be removed from the pulp.

[0022] The present invention complement the performance of bleaching pulp delignified at a high kappa so that it becomes industrially possible. Additionally, it allows pulp production to a significantly higher yield than in a conventional fiber line. Further, sulfur-poor lignin can be recovered from the process even though the pulp is produced by the sulphate method using a conventional fiber line and recovery concept. The invention is more preferably applied to soft wood in view of lignin amount and cooking/delignifying properties, but it can also be applied to hard wood, especially hard wood species having a high lignin-content.

[0023] In the presented method, the pulp is produced by an alkaline cooking method, such as a sulphate cook, a soda

cook or a soda-anthraquinone cook. The cooking method may also be modifications of these. A preferred example is a sulphate cook having a low sulfidity, i.e. 5-25%. Sulfidity values lower than that relate to soda cooking or modifications thereof. The cook may be a continuous cook or a batch cook.

[0024] In the cook, the pulp is cooked to a kappa number of 50-120, preferably 60-100. This kind of pulp has approximately 7-14 weight per cent lignin, which is an essentially significant amount. This lignin is removed in the oxygen stage so that the kappa number of the pulp will be typically approximately 20-40 and approximately 50, if the kappa of cooked pulp is 100-120. Thereby, a significant amount of lignin in view of both economical and process technology aspects is transferred into the oxygen stage filtrate. According to an embodiment of the invention, the oxygen stage is not carried out using oxidized white liquor, but preferably using sodium hydroxide, whereby the lignin is released in the solution, which is sulfur-poor compared to an oxygen stage carried out using oxidized white liquor. Sulfur from the magnesium sulphate used as an inhibitor and from cooking chemicals enters the oxygen stage. In this case, it is more advantageous to use in the washer upstream of the oxygen stage a liquid devoid of sulfur compounds generated from oxidized white liquor. Lignin that has less sulfur than normal is advantageous in many further use applications for lignin. The method according to the invention does not require or perform any separate chelate treatment prior to the oxygen stage. Thus it is possible to decrease the amount of effluent fractions and process costs.

[0025] After the oxygen stage, the pulp is washed normally so that reaction products are separated from the pulp in a liquid phase. It is possible to produce lignin in the amount of 40-120 kg, typically 40-80 kg, per ton of pulp. In the invention, the alkali used in the oxygen stage is preferably sulfur-free or sulfur-poor, whereby, when efficiently washing the pulp both downstream and upstream of the oxygen stage using liquid essentially free of sulfur compounds, it is possible to receive a lignin-containing liquid having sulfur-poor lignin in the amount of 40-100 kg per ton of pulp. The mon-etary value of this lignin is so significant that it is worth recovering, especially since leading lignin compounds to washing upstream of the oxygen stage is such a major disturbance factor for the process that it does not have process technical or economical grounds. Low-sulfur lignin refers to lignin having a sulfur-content less than 1% of the dry matter in the lignin. According to a study, the sulfur-content of precipitated from black liquor was approximately 1.4% (sulfur) of the dry matter and the sulfur-content of lignin precipitated from the oxygen stage washing filtrate was approximately 0.66% of dry matter.

[0026] In the solution presented herein, filtrate of the oxygen stage is separated from the pulp and lignin is recovered therefrom e.g. to be used as raw material for chemical industry. Correspondingly the liquid, wherefrom the lignin has been separated, can be re-used in the oxygen stage such that it is led to washing upstream of the oxygen stage, without harmful effect of lignin. Thus, removal oxygen reaction products of the oxygen stage from a counter-currently connected washing cycle and a separation technique that is in any case to be connected to the oxygen stage of high-kappa pulp can be regarded as investments having a clear economical target, and not only as additional cost for ensuring the functioning of the process.

[0027] In order to optimize the sulfur-content of lignin, the alkali of the oxygen stage is to be either pure sodium hydroxide (NaOH) or some other hydroxide that enables maintaining an adequately high pH-level in the oxygen stage. Pure here means that the sodium hydroxide solution does not contain any sulfur, or contains very small amounts of sulfur. Preferably the oxygen stage chemical is not a chemical that contains sulfur compounds. One chemical typically used in the oxygen stage is magnesium sulfate as an inhibitor. The use thereof can be optimized according to the pulp quality requirements and according to how low the sulfur-content is to be. Though oxidized white liquor is preferably not used, it is still possible in connection with chemical regeneration at a chemical pulp mill to separate the alkali so that one fraction contains the sulfur compounds and another the pure NaOH. In this case, the chemical proportion is formed so that the sulfidity in the cook is over 40%. The sulfidity returns to "normal" in the stream going to black liquor evaporation plant, when in accordance with the counter-current principle the cooked pulp is washed using low-sulfur washing liquid from lignin separation. In another embodiment the sulfidity of the cook is approximately 25-40%, but that of liquor in recovery it is clearly lower, e.g. 25-30%.

[0028] Prior art has presented several processes for fractionating qreen liquor to sodium hydroxide and sulfur -containing fractions in connection with chemical regeneration. This kind of processes are known e.g. from FI104334, FI98226 and US 5607549. The sodium hydroxide is used in the oxygen stage and the sulfur-containing fraction in the cook. Then there is no need to use purchased chemical as the alkali in the oxygen stage, but the required chemical can be produced at the mill in connection with chemical circulation.

[0029] According to an embodiment, also oxidized white liquor can be used in the oxygen stage. Then, in the production of white liquor, the sulfide is oxidized with oxygen via tiosulfate to sulfates. This is not a very advantageous embodiment, if minimizing the sulfur-content of the lignin is desired.

[0030] The lignin separation method as such is not essential in view of the invention, but before all the fact that lignin separation becomes profitable and that according to a preferred embodiment the lignin can in a kraft-process be separated in low-sulfur form. Then the process is to comprise the following properties:

Kraft-cook to kappa 50-120.

[0031] Efficient washing of pulp after cooking and the oxygen stage. The E10-value of the washing devices for cooked pulp at least 8, preferably over 10. The E10-value of the oxygen stage washer over 4, preferably over 7. The required washing efficiency is also dependent on how accurately the cook-generated lignin and the oxygen stage lignin are separated in the washes. The more efficient the washing of the cooked pulp, the better the cook-generated lingnin is separated and the more oxygen stage - generated lignin can be separated in the oxygen stage washing in low-sulfur form.

- The washing liquid introduced to the last stage of the wash upstream of the oxygen stage is liquid devoid of essential amounts of sulfur.
- The alkali during the oxygen stage is preferably sulfur-free, when very low-sulfur lignin is desired.
- In the oxygen stage the kappa is decreased by at least 30 units, preferably by more than 35 units, for releasing from the pulp an economically significant amount of lignin.

[0032] A significant portion of the lignin of the pulp is dissolved in the oxygen stage into the liquid traveling with the fibers and it is thus possible to separate from the filtrate of the washers or the presses. When the lignin has been separated, the thus purified liquid can be used in the washing of cooked pulp. The washed pulp is taken either to board production as such or it can be further bleached in a method presented also in this patent application.

[0033] Since a modern chemical pulp mill can already be almost totally closed as to sulfur and the mill is in practice free of sulfur losses, it is clear that the washing of cooked pulp can removed sulfur highly efficiently and thus a conventional washing technique with an E_{10}-value over 8, is suitable for removing sulfur from pulp.

[0034] Essential in connection with the present invention is that pulp that has been cooked to a significantly high lignin-content can be processed for the market to pulp that can also be washed to a desired cleanliness level. This opens a totally new possibility for building a biorefinery and applying various treatment methods, as a result of which lignin can be separated from fiber slurry. Naturally, possible embodiments are often limited to very affordable chemicals, because the process is to be made profitable, but still several various methods exist that can be applied for lignin separation.

[0035] Separated lignin can also be used as fuel in the boilers and kilns of a chemical pulp mill.

[0036] During the last years, the wood processing industry and also the chemical industry have awakened to search substitute raw materials for producing polymers and other carbon-based materials. At present, the most important raw material of polymer industry is crude oil, the price and availability of which in the long run will make wood-based raw materials for polymers competitive. Thus, also lignin based on a chemical pulping process has market where it can replace crude oil -based products. As the most important chemical pulp production processes are based on the use sulfur-based cooking chemicals, the usability of lignin separated from those is in the chemical industry low or restricted, if sulfur-removal from lignin is not taken care of. And if the cooking process is accomplished using sulfur-free chemicals, the quality of lignin is improved, but the pulp production assumes many such features that the processes have not yet become widely competitive. An advantage of the present invention is that it produces lignin suitable of further processing.

[0037] The oxygen stage is the simplest and best known method for delignifying high kappa pulp. Then the pulp is treated with a method in which fiber properties are the priority and the objects of use of the fiber raw material are known paper and fiber products. It is also possible to use other lignin dissolving and separation method, replacing the oxygen stage, which are used to produce e.g. special lignins or to separate e.g. hemicelluloses. This kind of methods could comprise an acid pulping method, such as a defibration suitable for grass pulp presented by Chempolis, a formic acid pulping known as the CIMV-process, or a combination of acid hydrolysis and an oxygen stage. In this relation, there are several alternatives, since high kappa pulp (kappa e,g, 100), from where cooking chemicals, first of all sulfur, have been removed by washing, is raw material for numerous different products. Thus, lignin is not the only possible product, but in addition to it, the products may comprise e.g. hemicelluloses.

[0038] A solution that is separated from the pulp in the oxygen stage can be treated in many ways for separating lignin. The most conventional chemical separation method is precipitation, where an alkaline solution is neutralized or acidified, whereby alkali is precipitated from the solution and it can be clarified out of the liquid. This requires an abundant amount of acid, but on the other hand it is an efficient and simple way of recovering lignin. About 15% of the lignin is precipitated at a pH of 10 and 50% at a pH of 8. The pH of the oxygen stage is 10-13, typically 10.5-12. In lignin separation there is no need to aim at complete separation. A satisfactory result is obtained in the washing of cooked pulp even if only approximately 50% of the lignin is separated from the oxygen stage washing filtrate prior to using it as washing liquid. On the other hand, circulating lignin via the oxygen stage deteriorates its quality so that it is advantageous to aim at a higher separation degree. Greater amount of native lignin in the recovered lignin is advantageous in view of further processing.

[0039] Other lignin separation ways comprise various filtration methods, in which a filter can remove large chained molecules, but lets ions and water go through. This kind of methods comprise various ultra- or nanofiltrations, osmosis and reverse osmosis, dynamic cross flow -filtration, and other methods by means of which molecules can be separated from a solution. When the lignin has been separated from the solution, the cleaned solution can be utilized in the washing of cooked pulp, because the organic loading has been removed.

[0040] When looking at a complex, where this kind of solution is suitable, such advantages show up, which as individual steps introduce remarkable improvements in the economical potential of the mill:

- The pulp yield of the fiber line is clearly better.
- A solution is obtained by means of which the oxygen stage functions well when high kappa pulp is treated.
- A process is formed where separation of low-sulfur lignin is possible.
- The whole chemical cycle of the process, such as evaporation and the recovery boiler, remain unchanged, so that the biorefinery solution possibly required for further use of the lignin does not require new process department.
- Greater production with a smaller chemical cycle. When lignin is recovered from the process, the amount of substance to be combusted in the recovery boiler is decreased, because the produced dry-matter amount per ton of pulp is lower, and the chemical cycle is smaller. Then, a smaller boiler is needed, or the existing boiler can burn a higher amount of black liquor dry substance per ton of produced pulp.

[0041] Since all changes have an influence on many units, the following type of matters are to be taken into account in developing the process:

- Overall steam production of the recovery boiler is decreased
- A new separation department for lignin is needed
- Chemicals of the oxygen stage or other delignifying stage may be more expensive than the presently used
- Effluent production changes and the conventional coupling can not be used as such
- There will be a need for sulfur-free or low-sulfur chemicals, the price level of which is higher than that of sulfur-containing chemicals.

[0042] The invention is especially applicable to pulp that is produced by a sulfate method or its modification. The use of e.g. yield enhancing chemicals, such as anthraquinone, is possible in connection with cooking. The pulp is cooked in a continuous or a batch cook so that the delignification is terminated at a high lignin-content level (kappa number 50-120); but so that the defiberization point of the chips is reached. This kidn of method is described e.g. in WO Patent Application PCT/US10/57417. The present method comprises as a preferred embodiment a process coupling, in which the mechanical defibration of the complete pulp stream after cooking can be omitted. Thus, the cooked pulp is discharged from the digester into washing and then into the oxygen stage without mechanical treatment in a mechanical defibrating device, e.g. a blow line refiner. In a mechanical defibrator, mechanical work is applied for releasing fibers from a chips matrix, which in the method according to this embodiment is not needed. The fiber line naturally comprises pumps, mixers and corresponding devices that are used for pulp transfer and for mixing chemicals and other substances into the pulp. Eliminating the mechanical defibrator is advantageous in view of energy economics, since the energy dissipation of the washer, pumps and mixers between the digester and the first oxygen stage is below 30 kW/adt, while a defibrator doubles that.

[0043] Screening of the pulp is more preferably accomplished after the oxygen delignification. According to an embodiment, the screen room is located downstream of the first oxygen stage, when there are several oxygen stages. According to an embodiment, the screen room is located between the first oxygen stage and the subsequent wash. Alternatively, the pulp can be screened also later, but preferably prior to washing following the second oxygen stage. The screening can also be divided into coarse screening and fine screening, whereby the coarse screening takes place e.g. downstream of the first oxygen stage and the fine screening downstream of the second oxygen stage. Thus, for producing pulp to be bleached it is advantageous to merely wash the pulp after cooking prior to the first oxygen stage. Thus, the pulp discharged from the digester is led, without mechanical defibration and without screening, via washing into the first oxygen stage.

[0044] However, it is conceivable that knot removal from the pulp is performed after discharging the pulp from the digester prior to the first wash. Thereby, in screening a knot reject separated from the main pulp stream is subjected to a gentle defibration. The defiberized reject stream can be returned to the main pulp stream either to the knot separation feed or co-currently downstream of knot separation. It is essential that the main pulp stream is not defibrated mechanically, but only a side stream separated therefrom (e.g. preferably less than 30%, most preferably less than 20%) is defibrated such that the knot fraction is in a suitable condition for washing and subsequent treatment.

[0045] Washing refers to one or more washing stages and thus it takes place in one or more washing apparatuses.

[0046] According to an embodiment, the pulp discharged from the digester is washed prior to the first oxygen stage, preferably using a single-drum washer, such as a pressure drum washer produced by Andritz, i.e. a Drum Displacer™ (DD)-washer, or a vacuum drum washer or a diffuser. A washer, in which the pulp gets pressed between drums and/or rolls in a so-called nip found e.g. in washing presses, is not suitable for pulp washing between the cook and the first oxygen stage. If knot removal is effected prior to washing downstream of the digester, the use of a press is possible, too. Washing downstream of the first oxygen stage can be accomplished using conventional washing technique, which

enables using the above mentioned washing apparatuses, and thus also a washing press or a press, when screening has taken place prior to the oxygen stage washing.

[0047] The first oxygen stage is a clear continuation to cooking, the purpose of which is to lower the kappa by 30-70 kappa units. Simultaneously also the reject content of the pulp decreases considerably, whereby in pulp screening at some treatment step after the first oxygen stage no remarkable amounts of reject are removed, since removal would end the yield benefit. In the second oxygen stage the kappa number of the pulp is lowered to a level of 10-20, preferably to a value of 10-15 or less.

[0048] Studies have revealed that the reject content in the first oxygen stage decreases even more than 95%, meaning that this coupling is especially efficient in lowering the reject content.

[0049] It is known that an oxygen molecule can only react with certain, i.e. phenolic lignin compositions. Under highly alkaline conditions (pH>10), an ionized lignin composition is decomposed and diminished as a result of chain reaction, and dissolved. Oxygen reacts primarily with free phenolic hydroxyl groups of lignin forming carboxyl groups, which convert the lignin to water-soluble form. Simultaneously, depolymerization, i.e. degradation, of lignin takes place. These reactions lead to decrease in the number of phenolic hydroxyl groups of lignin and increase in the number of carboxyl groups of lignin.

[0050] This phenolic lignin is removed in oxygen delignification in accordance with oxygen delignification kinetics, but oxygen delignification does not have any notable effect on the amount of non-phenolic lignin. For this chemistry, the delignification level in the oxygen stage stops at a level of 75-80%, even if oxygen treatments with the same chemical conditions were coupled several in series and the treatment period continued. Additionally, harshening of the conditions is clearly seen, above all as decreased quality of pulp and low yield. Thus, continuing delignification after the oxygen stage requires chemicals that also react with other lignin compositions and /or create new free phenolic groups.

[0051] Experiments have also been made to find out whether the treatment of pulp under acid conditions would activate the oxygen stage adequately, but it was noticed that an acid stage as such, without reaction with lignin, did not help in lowering the kappa. An acid stage intensifies mass transfer, but does not change the chemical composition. Thus, it has been discovered that after the oxygen stage the pulp is to be subjected to treatment with a bleaching chemical that reacts with non-phenolic lignin. Suitable for this are so-called electrophilic bleaching chemicals. These typically comprise peracids, chlorine dioxide under acid conditions, ozone or chlorine. Peracids comprise e.g. peracetic acid, Caron acid, persulphuric acid, and peroxomolyb-dates. Treatment of pulp with these chemicals can be performed under conditions that are known per se and presented in handbooks for the field. Chlorine dioxide treatment is typically performed at a temperature of 40 - 90 °C, at a pH of 1.5-5.5 and during 1-10 minutes, but longer periods are also possible. A typical chlorine dioxide dose in the treatment is 2 - 20 kg/adt. Typical conditions for peracid treatments are: temperature 50-90 °C and time 30-120 minutes. The amount of chemical added into the pulp depends on the peracid used, but typically it is 2-20 kg/adt.

[0052] Treatment with ozone is typically effected pressurized under a pressure of 5-15 bar, preferably at a consistency of 5-35%, more preferably at a consistency of 7-18%, so that ozone-containing gas having an ozone-content of 8-18 weight per cent, preferably 10-15%, is introduced into the pulp at one or several points, preferably via one or more mixers. Said gas is a mixture of ozone and oxygen that acts as carrier gas. The amount of ozone dosed into the pulp is 2 - 8 kg/admt. The pH is in the acid range, typically 1-5. Ozone bleaching is performed either only in mixers allowing a long retention or so that the mixture of pulp and ozone with carrier gas is introduced into a reaction vessel, wherein the desired reaction retention is arranged.

[0053] After electrophilic chemical treatment, the pulp is delignified a second time with oxygen, the purpose of which is lowering the kappa number to a targeted kappa number of 10-20 or in some cases even lower than 10. After this the pulp can be bleached in a conventional total chlorine free (TCF) bleaching process or in an elementary chlorine free (ECF) bleaching process.

[0054] The present invention is described in more detail by means of an embodiment according to the invention and with reference to the accompanying schematic drawings, in which:

Fig. is a schematic illustration of a preferred oxygen delignification of high kappa pulp without lignin separation; and Figure 2 is a schematic illustration of a preferred exemplary embodiment of the method according to the present invention.

[0055] The digester equipment comprises one or more pulp digesters. Wood pulp cooked to a kappa number of 50-120, typically 60-100, is taken from the bottom of the digester 1 via blow line 2 into a blow tank 3, from where the pulp is pumped via line 4 into a washer 5. The washer is typically a single drum washer, such as e.g., a pressure drum washer produced by Andritz, i.e. a Drum Displacer™ (DD)-washer, or a diffuser or a suction drum washer. The washed pulp is led into an oxygen stage. The pulp discharged from the digester is not defibrated mechanically and therefore the blow line is devoid of a mechanical defibrator downstream of the digester. Also, the pulp is not screened here prior to the first oxygen stage. The blow tank 3 is not inevitable, but the pulp can also be taken directly into the washer 5, depending on

the type of washer and equipment coupling, as known per se.

[0056] Black liquor is discharged from the digester via line 33.

[0057] In this example, the first oxygen stage comprises two reactors 7 and 8, i.e. it is a two-step stage, but it can also be a one-step or multi-step stage, as described above. In this oxygen stage the kappa number of the pulp typically decreases by 30-70 units.

[0058] From the first oxygen stage the pulp is led via line 9 to a screening room 10. The pulp is led further via line 11 to washing 12 downstream of the oxygen stage, where the washer can instead of a single drum washer or a diffuser be also a press.

[0059] Next the pulp is treated with an electrophilic chemical in a reaction vessel 14, wherein the washed pulp is discharged from the washing apparatus 12 via line 13. The pulp is treated after the first oxygen stage with such a bleaching chemical that reacts with non-phenolic lignin. Suitable for this are so-called electrophilic bleaching chemicals. These typically comprise peracids, chlorine dioxide under acid conditions, ozone or chlorine. This treatment adequately promotes the delignifiction of pulp cooked to a high kappa for the actual bleaching process.

[0060] After treatment in the reaction vessel 14 the pulp is discharged via line 15 into a washer 16, which can be a similar apparatus as the washer 12. After this the washed pulp is taken via line 17 to a second oxygen stage, which similar to the first oxygen stage comprises two steps 18 and 19. In this oxygen stage the kappa number of the pulp is lowered to a level of 10-20, preferably 10-15 or less.

[0061] The pulp from the second oxygen stage is led via line 20 into a washer 21, which can be single drum washer or a diffuser or also a press. The washed pulp is taken via line 27 into a bleaching process. The pulp that has been cooked to a high kappa has after the cook been treated so that its kappa number has been decreased to a level advantageous for bleaching, but simultaneously the quality of the pulp has remained at a good level, since no mechanical defibration has been needed.

[0062] Figure 1 illustrates that the pulp is washed as known per se in accordance with counter-current principle. Thereby the filtrate from the latter oxygen stage washing 21 is led counter-currently via line 22 into a washer 16 as washing liquid. Because treatment 14 with an electrophilic chemical is carried out under acid conditions, a portion of the filtrate from the downstream washer is discharged from the process via line 24, but a portion is taken via line 23 to the first oxygen stage washer 12 as washing liquid. The filtrate discharged from the washer 12 is led via line 28 to a washer 5 downstream of the digester, the filtrate from which washer is led via line 26 to digester washing into digester 1. Additionally, water or other washing liquid is introduced into the washers 21 and 12 via lines 32.

[0063] In connection with Figure 2, the same reference numerals as in figure 1 have been used where applicable. Wood pulp cooked to a kappa number of 50-120, typically 60-100, is taken from the bottom of the digester 1 via blow line 2 into a blow tank 3, from where the pulp is pumped via line 4 into one or several washers 5. The washer is typically a single drum washer, such as e.g., a pressure drum washer produced by Andritz, i.e. a Drum Displacer™ (DD)-washer, or a diffuser or a suction drum washer. Before that, the cooked pulp is washed at the bottom of the digester by a so-called digester wash. The E_{10}-value of the washing equipment for cooked pulp, i.e. digester wash and the washer 5, is at least 8, preferably at least 10.

[0064] The washed pulp is led into an oxygen stage. In this embodiment, the first and the second oxygen stage have one reaction vessel 8 and 19, respectively, but there may be more vessels, as in Figure 1. A further difference compared to the arrangement of Figure 1 is the treatment of the filtrate of the washer downstream of the oxygen stage. In the oxygen stage, especially in the first treatment, a large amount of lignin is released. When the amount of removed lignin is e.g. 40 kappa units, the amount of organic material being released from the pulp becomes high. With said kappa reduction, the proportion of e.g. lignin only is approximately 7% of the dry matter of the pulp, and thereto carbohydrate losses. Due to this, the COD-amount of the solution, which characterizes the organic substance in the filtrates, may rise to 100-200 kg/adt as a result of lignin. In Figure 2, the filtrate of the washer 12 downstream of the first oxygen stage 8 contains abundantly lignin, especially if the kappa number in the reaction vessel 8 has been decreased by 30-70 units. Therefore, lignin is separated from the filtrate 25 of the washer 12 in stage 29 by a method that may be known as such, e.g. by precipitating or filtering. Various lignin separation methods can be applied here. It is advantageous that the first oxygen treatment uses sodium hydroxide as alkali instead of oxidized white liquor for avoiding sulfur compounds. The lignin fraction is recovered in low-sulfur form, and thus it is advantageously used as raw material for chemical industry. As a result of lignin separation, the filtrate fluid is cleaned, and the filtrate is used at the washer 5 downstream of the digester and upstream of the oxygen treatment, from which washer the filtrate is taken via line 31 into digester washing. The lignin-containing filtrate 25 is in Figure 1 used as washing liquid for pulp at the washer 5 without separating lignin.

[0065] In the following, the results of a series of experiments are shown in form of a table, in which the pulp was treated in connection with the oxygen stage with various sequences. The experiments were conducted for Scandinavian soft wood pulp (SW). From the data in the table it is noticed that pulp treated with either acid peroxide (Caron acid) or chlorine dioxide has been delignified to a kappa number level of approximately 15. The kappa number of pulps treated with hydrochlorite or acid only or not treated at all, was, in turn, at a level of 20. The results confirm the fact that the kappa reduction of high kappa pulp in the oxygen stage is at the most approximately 80%. For re-activating the pulp for the

oxygen stage, a treatment with an electrophilic chemical (Caron acid or chlorine dioxide) is required. This kind of pulp can be bleached to high brightness.

Initial pulp	Kappa number	-	79,1				
	Brightness	% ISO	20,4				
	Reject content	% (on pulp)	28,7				
	Sequence		**O-A-O**	**O-Px(H_2SO_5)-O**	**O-D_0-O**	**O-H-O**	**O-O_P**
1st Oxygen delignification stage	Reactor type	-	MC-mixer	MC-mixer	MC-mixer	MC-mixer	MC-mixer
	Consistency	%	10	10	10	10	10
	Temperature	°C	100	100	100	100	100
	Retention time	min	10 / 50	10 / 50	10 / 50	10 / 50	10 / 50
	O_2-pressure	bar(g)	6.9 / 5.5	6.9 / 5.5	6.9 / 5.5	6.9 / 5.5	6.9 / 5.5
	NaOH-charge	% (on pulp)	6,5	6,5	6,5	6,5	6,5
	$MgSO_4$-charge	% (on pulp)	0,15	0,15	0,15	0,15	0,15
	Final pH	-	11,8	11,1	11,4	11,4	11,2
	Kappa number	-	32,8	31,7	31,9	29,6	30,7
	Viscosity	mL/g	1020	1030	1031	1011	1003
	Brightness	% ISO	28,6	29,5	30,3	31	29,1
	Yield	% (on pulp)	89,59	89,91	88,8	89,9	88,1
	Reject content	% (on pulp)	0,08	0,12	0,08	0,04	0,08
	COD	kg/BDMTp	na	164,6	175,4	183,3	191,2
Intermediate activation stage	Type		A	Px	D_0	H	-
	Consistency	%	10	10	10	10	-
	Temperature	°C	60	70	70	70	-
	Retention time	min	60	60	15	30	-
	Chemical charge	% (on pulp)	H_2SO_4 0.95	H_2SO_5 2.0	ClO_2 0.38	NaOCl 0.5	-
	Chemical charge	% (on pulp)	-	NaOH 4.9	H_2SO_4 0.88	NaOH 0.69	-
	Final pH	-	3,5	2,9	2,9	11,2	-
	Kappa number	-	na	26,7	28,5	28,3	-
	Viscosity	mL/g	na	877	na	1018	-
	Brightness	% ISO	na	37,8	na	36,4	-
2nd Oxygen delignification stage	Reactor type		Autoclave	Autoclave	Autoclave	Autoclave	Autoclave
	Consistency	%	10	10	10	10	10
	Temperature	°C	100	100	100	100	100
	Retention time	min	85	85	85	85	85
	O_2-pressure	bar(g)	5,5	5,5	5,5	5,5	5,5
	NaOH-charge	% (on pulp)	3,5	3,5	3,5	3,5	3,5
	$MgSO_4$-charge	% (on pulp)	0,15	0,15	0,15	0,15	0,15
	H_2O_2-charge	% (on pulp)	-	-	-	-	0,6
	Final pH	-	11,5	11,5	11,6	12	11,9
	Kappa number	-	20,8	14,8	16,2	18,8	18,9
	Viscosity	mL/g	900	791	915	892	856
	Brightness	% ISO	37,2	48,2	47,1	37,2	40,9
	Yield	% (on pulp)	96,6	96,5	95,9	96,2	97,5
	COD	kg/BDMTp	na	76,5	59,8	54,3	67,6

[0066] The present method can provide an efficient washing and delignifying method for producing pulp from the high kappa pulp so that the quality of the pulp is not deteriorated and that the yield is good.

[0067] Although the above description relates to an embodiment of the invention that is in the light of present knowledge considered the most preferable, it is obvious to a person skilled in the art that the invention can be modified in many different ways within the broadest possible scope defined by the appended claims alone.

Claims

1. A method of treating chemical pulp, in which method

pulp is produced by cooking wood chips in an alkaline cooking process to a kappa number of 50-120, preferably 60-100,
the cooked pulp is washed,
the washed pulp is led into an oxygen stage where the pulp is treated in the presence of oxygen and alkali for removing lignin, and the oxygen-treated pulp is washed, whereby a lignin-containing washing filtrate (25) is formed, **characterized in that**
in the oxygen stage the kappa number of the pulp decreases by at least 30 units, and
the filtrate (25) of the washing downstream of the oxygen stage is treated for separating lignin, and the treated filtrate is led as washing liquid into the washing apparatus (5) for the cooked pulp.

2. A method according to claim 1, **characterized in that** the E_{10}-value of the washing apparatus (5) for the cooked pulp is at least 8, preferably at least 10.

3. A method according to claim 1 or 2, **characterized in that** the in the oxygen stage the kappa number of the pulp decreases by at least 35 kappa units.

4. A method according to claim 1, 2 or 3, **characterized in that** during the oxygen stage the alkali is sodium hydroxide.

5. A method according to any of the preceding claims, **characterized in that** the pulp is not screened prior to the oxygen stage, but after that.

6. A method according to claim 5, **characterized in that** the pulp is treated in two oxygen stages and screened between the first oxygen stage and the washing downstream of the second oxygen stage.

7. A method according to any of the preceding claims, **characterized in that** it comprises knot-separation prior to treating the pulp with oxygen.

8. A method according to any of the preceding claims, **characterized in that** the lignin is separated by precipitating.

9. A method according to any of the preceding claims, **characterized in that** the cooking process is a sulfate process.

10. A method according to any of the preceding claims 1-8, **characterized in that** the cooking process is a soda process or a modification thereof.

11. A method according to any of the preceding claims, **characterized in that** the pulp is discharged from the digester (1) and led to washing without treatment in a mechanical defibrator between the digester (1) and the washing.

12. A method according to any of the preceding claims, **characterized in that** the separated lignin is recovered and used as such or further processed.

13. An arrangement for treating pulp produced from wood chips in a cooking apparatus (1) wherein the wood chips are cooked chemically to a kappa number of 50-120, preferably 60-100, said arrangement comprising at least

a cooking apparatus (1) for cellulosic fibrous material,
washing devices (5) for cooked pulp,
apparatuses (8) downstream of washing of the cooked pulp for delignifying the pulp in an oxygen stage,
apparatuses (12) for washing the pulp after the oxygen stage, and

filtrate lines (28, 31) for leading washing filtrates counter-currently to preceding washers (5) as washing liquid, **characterized in that** the filtrate line (28) down
stream of the oxygen stage is provided with devices (29) for separating lignin from the filtrate (25), and the filtrate line (28) is connected to the washing devices (5) for cooked pulp for using the filtrate as washing liquid after lignin separation.

14. An arrangement according to claim 13, **characterized in that** it comprises

a washing apparatus (5), a pulp inlet conduit (4) of which is connected to a pulp outlet conduit (2) of the cooking apparatus (1) and the pulp outlet conduit is connected to a pulp inlet conduit (6) of the oxygen-treatment apparatuses (8), and
a washing apparatus (12) subsequent to the oxygen-treatment apparatuses (8), a pulp inlet conduit (11) of which is connected to the pulp outlet conduit (9) of the oxygen-treatment apparatuses (8) for leading the pulp into the washing apparatus (12), whereby the lignin-separation devices (29) are arranged in a filtrate line (28) between the washing apparatuses (5, 12).

15. An arrangement according to claim 13 or 14, **characterized in that** the E_{10}-value of the washing equipment (5) for the cooked pulp is at least 8, preferably at least 10.

Patentansprüche

1. Verfahren zum Behandeln von chemischem Zellstoff, wobei in dem Verfahren
Zellstoff durch Kochen von Holzschnitzeln in einem alkalischen Kochverfahren zu einer Kappazahl von 50-120, vorzugsweise 60-100, hergestellt wird,
der gekochte Zellstoff gewaschen wird,
der gewaschene Zellstoff in eine Sauerstoffstufe geführt wird, wobei der Zellstoff in Gegenwart von Sauerstoff und Alkali zum Entfernen von Lignin behandelt wird, und der Sauerstoff-behandelte Zellstoff gewaschen wird, wodurch ein Lignin-enthaltendes Waschfiltrat (25) gebildet wird, **dadurch gekennzeichnet, dass**
in der Sauerstoffstufe die Kappazahl des Zellstoffs um mindestens 30 Einheiten gesenkt wird, und
das Filtrat (25) der Waschung stromabwärts der Sauerstoffstufe zum Trennen von Lignin behandelt wird, und das behandelte Filtrat als Waschflüssigkeit in die Wascheinrichtung (5) für den gekochten Zellstoff geführt wird.

2. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, dass** der E_{10}-Wert der Wascheinrichtung (5) für den gekochten Zellstoff mindestens 8, vorzugsweise mindestens 10 ist.

3. Verfahren nach Anspruch 1 oder 2, **dadurch gekennzeichnet, dass** in der Sauerstoffstufe die Kappazahl des Zellstoffs um mindestens 35 Kappa-Einheiten sinkt.

4. Verfahren nach Anspruch 1, 2 oder 3, **dadurch gekennzeichnet, dass** während der Sauerstoffstufe das Alkali Natriumhydroxid ist.

5. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, dass** der Zellstoff vor der Sauerstoffstufe nicht sortiert wird, sondern danach.

6. Verfahren nach Anspruch 5, **dadurch gekennzeichnet, dass** der Zellstoff in zwei Sauerstoffstufen behandelt und zwischen der ersten Sauerstoffstufe und dem Waschen stromabwärts von der zweiten Sauerstoffstufe sortiert wird.

7. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, dass** es Astabscheidung vor dem Behandeln des Zellstoffs mit Sauerstoff umfasst.

8. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, dass** das Lignin durch Ausfällung abgetrennt wird.

9. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, dass** das Kochverfahren ein Sulfat-Verfahren ist.

10. Verfahren nach einem der vorstehenden Ansprüche 1-8, **dadurch gekennzeichnet, dass** das Kochverfahren ein

Soda-Verfahren oder eine Modifizierung davon ist.

11. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, dass** der Zellstoff von dem Zellstoffkocher (1) ausgegeben wird und zum Waschen ohne Behandlung in einem mechanischen Zerfaserer zwischen dem Zellstoffkocher (1) und dem Waschen geführt wird.

12. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, dass** das abgetrennte Lignin gewonnen und als solches verwendet oder weiter verarbeitet wird.

13. Anlage zur Behandlung von Zellstoff, hergestellt aus Holzschnitzeln in einer Koch-Einrichtung (1), wobei die Holzschnitzel chemisch zu einer Kappazahl von 50-120, vorzugsweise 60-100, gekocht werden, wobei die Anlage mindestens umfasst
eine Koch-Einrichtung (1) für Zellulosefasermaterial,
Waschvorrichtungen (5) für gekochten Zellstoff,
Einrichtungen (8) stromabwärts des Waschens des gekochten Zellstoffs zum des Zellstoffs in einer Sauerstoffstufe,
Einrichtungen (12) zum Waschen des Zellstoffs nach der Sauerstoffstufe und Filtratleitungen (28, 31) zum Führen der Waschfiltrate im Gegenstrom zu vorangehenden Wäschern (5) als Waschflüssigkeit, **dadurch gekennzeichnet, dass** die Filtratleitung (28) stromabwärts der Sauerstoffstufe mit Vorrichtungen (29) zum Trennen von Lignin von dem Filtrat (25) ausgestattet ist, und die Filtratleitung (28) mit den Waschvorrichtungen (5) für gekochten Zellstoff zum Verwenden des Filtrats als Waschflüssigkeit nach Ligninabtrennung verbunden ist.

14. Anlage nach Anspruch 13, **dadurch gekennzeichnet, dass** sie umfasst
eine Wascheinrichtung (5), deren Zellstoff-Einlassleitungsrohr (4) mit einem Zellstoff-Auslassleitungsrohr (2) der Koch-Einrichtung (1) verbunden ist, und wobei das Zellstoff-Auslassleitungsrohr mit einem Zellstoff-Einlassleitungsrohr (6) der Sauerstoff-Behandlungs-Einrichtungen (8) verbunden ist, und
eine Wascheinrichtung (12) anschließend an die Sauerstoff-Behandlungs-Einrichtungen (8), deren Zellstoff-Einlassleitungsrohr (11) mit dem Zellstoff-Auslassleitungsrohr (9) der Sauerstoff-Behandlungs-Einrichtungen (8) zum Führen des Zellstoffs in die Wascheinrichtung (12) verbunden ist, wodurch die Lignin-TrennVorrichtungen (29) in einer Filtratleitung (28) zwischen den Wascheinrichtungen (5, 12) angeordnet sind.

15. Anlage nach Anspruch 13 oder 14, **dadurch gekennzeichnet, dass** der E_{10}-Wert der Wasch-Ausrüstung (5) für den gekochten Zellstoff mindestens 8, vorzugsweise mindestens 10 ist.

Revendications

1. Procédé de traitement d'une pâte chimique, procédé dans lequel :

de la pâte est produite par cuisson de copeaux de bois dans un processus de cuisson alcalin pour obtenir un indice kappa de 50 à 120, de préférence de 60 à 100 ;
la pâte cuite est lavée;
la pâte lavée est conduite à une étape de traitement à l'oxygène dans laquelle la pâte est traitée en présence d'oxygène et d'alcali pour éliminer la lignine, et la pâte traitée à l'oxygène est lavée, donnant lieu à la formation d'un filtrat de lavage (25) contenant de la lignine, **caractérisé en ce que** :

dans l'étape de traitement à l'oxygène, l'indice kappa de la pâte diminue à concurrence d'au moins 30 unités; et
le filtrat (25) du lavage en aval de l'étape de traitement à l'oxygène est traité pour séparer la lignine, et le filtrat traité est conduit sous la forme d'un liquide de lavage dans l'appareil de lavage (5) pour la pâte cuite.

2. Procédé selon la revendication 1, **caractérisé en ce que** la valeur E_{10} de l'appareil de lavage (5) pour la pâte cuite s'élève à au moins 8, de préférence à au moins 10.

3. Procédé selon la revendication 1 ou 2, **caractérisé en ce que** dans l'étape de traitement à l'oxygène, l'indice kappa de la pâte diminue à concurrence d'au moins 35 unités.

4. Procédé selon la revendication 1, 2 ou 3, **caractérisé en ce que**, au cours de l'étape de traitement à l'oxygène, l'alcali est de l'hydroxyde de sodium.

5. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que** la pâte n'est pas soumise à un classage avant l'étape de traitement à l'oxygène, mais après ladite étape.

6. Procédé selon la revendication 5, **caractérisé en ce que** la pâte est traitée dans deux étapes de traitement à l'oxygène et est classée entre la première étape de traitement à l'oxygène et le lavage en aval de la seconde étape de traitement à l'oxygène.

7. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce qu'**il comprend une séparation des noeuds avant le traitement de la pâte avec de l'oxygène.

8. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que** la lignine est séparée par précipitation.

9. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que** le processus de cuisson est un processus faisant appel à du sulfate.

10. Procédé selon l'une quelconque des revendications 1 à 8, **caractérisé en ce que** le processus de cuisson est un processus faisant appel à de la soude, ou une de ses modifications.

11. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que** la pâte est évacuée du lessiveur (1) et conduite à un lavage sans traitement dans un dispositif mécanique de défibrage entre le lessiveur (1) et le lavage.

12. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que** la lignine séparée est récupérée et est utilisée comme telle ou est soumise à un traitement ultérieur.

13. Arrangement pour traiter de la pâte produite à partir de copeaux de bois dans un appareil de cuisson (1) dans lequel les copeaux de bois sont soumis à une cuisson chimique pour obtenir un indice kappa de 50 à 120, de préférence de 60 à 100, ledit arrangement comprenant au moins :

un appareil de cuisson (1) pour une matière fibreuse cellulosique,
des dispositifs de lavage (5) pour la pâte cuite,
des appareils (8) en aval du lavage de la pâte cuite pour la délignification de la pâte dans une étape de traitement à l'oxygène,
des appareils (12) pour laver la pâte après l'étape de traitement à l'oxygène, et
des lignes pour le filtrat (28, 31) pour conduire les filtrats de lavage à contre-courant par rapport aux lessiveurs (5) qui précèdent, à titre de liquide de lavage, **caractérisé en ce que** la ligne pour le filtrat (28) en aval de l'étape de traitement à l'oxygène est équipée de dispositifs (29) pour séparer la lignine du filtrat (25), et la ligne pour le filtrat (28) est raccordée aux dispositifs de lavage (5) pour la pâte cuite afin d'utiliser le filtrat comme liquide de lavage après la séparation de la lignine.

14. Arrangement selon la revendication 13, **caractérisé en ce qu'**il comprend :

un appareil de lavage (5), dont un conduit d'entrée pour la pâte (4) qui est raccordé à un conduit de sortie pour la pâte (2) de l'appareil de cuisson (1) et le conduit de sortie pour la pâte est raccordé à un conduit d'entrée pour la pâte (6) des appareils de traitement à l'oxygène (8), et
un appareil de lavage (12) qui fait suite aux appareils de traitement à l'oxygène (8), dont un conduit d'entrée pour la pâte (11) est raccordé au conduit de sortie pour la pâte (9) des appareils de traitement à l'oxygène (8) pour conduire la pâte jusque dans l'appareil de lavage (12), les dispositifs de séparation de la lignine (29) étant disposés dans une ligne pour le filtrat (28) entre les appareils de lavage (5, 12).

15. Arrangement selon la revendication 13 ou 14, **caractérisé en ce que** la valeur E_{10} de l'équipement de lavage (5) pour la pâte cuite s'élève à au moins 8, de préférence à au moins 10.

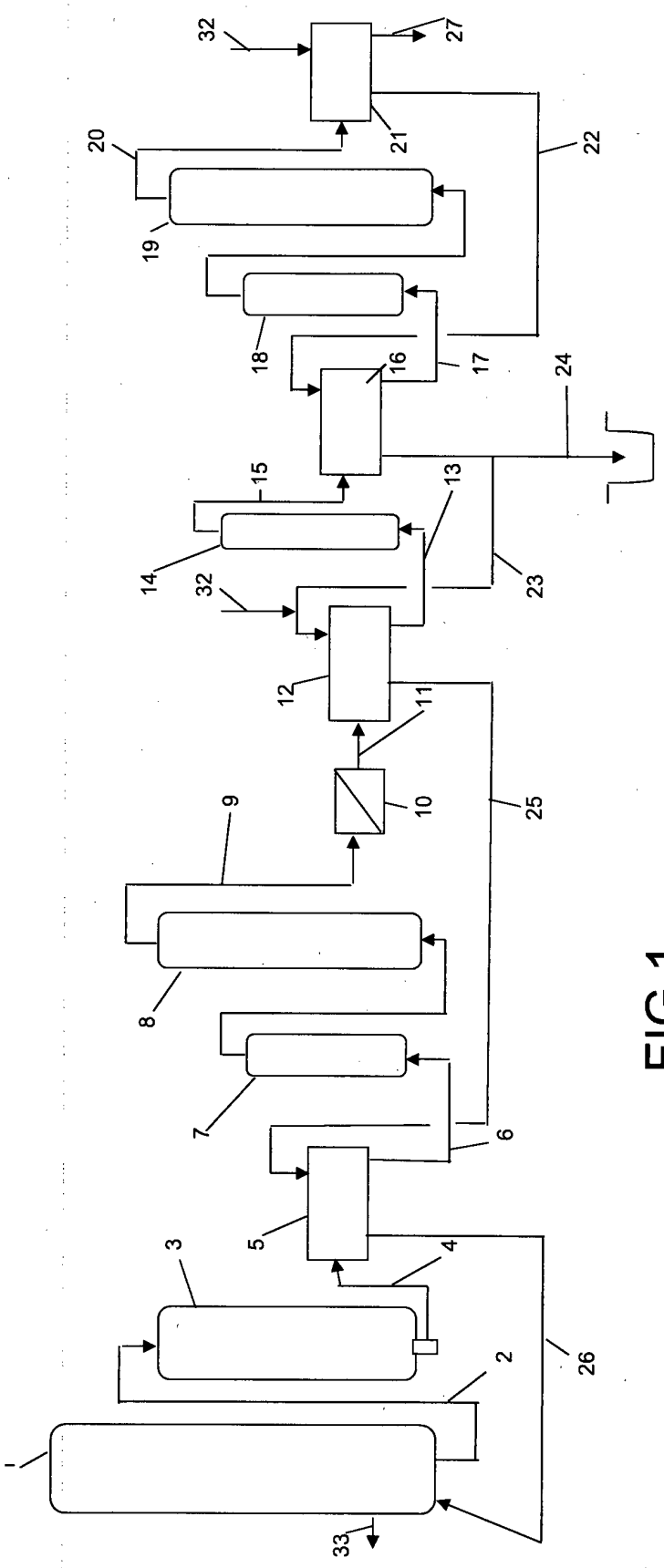

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FIG.1

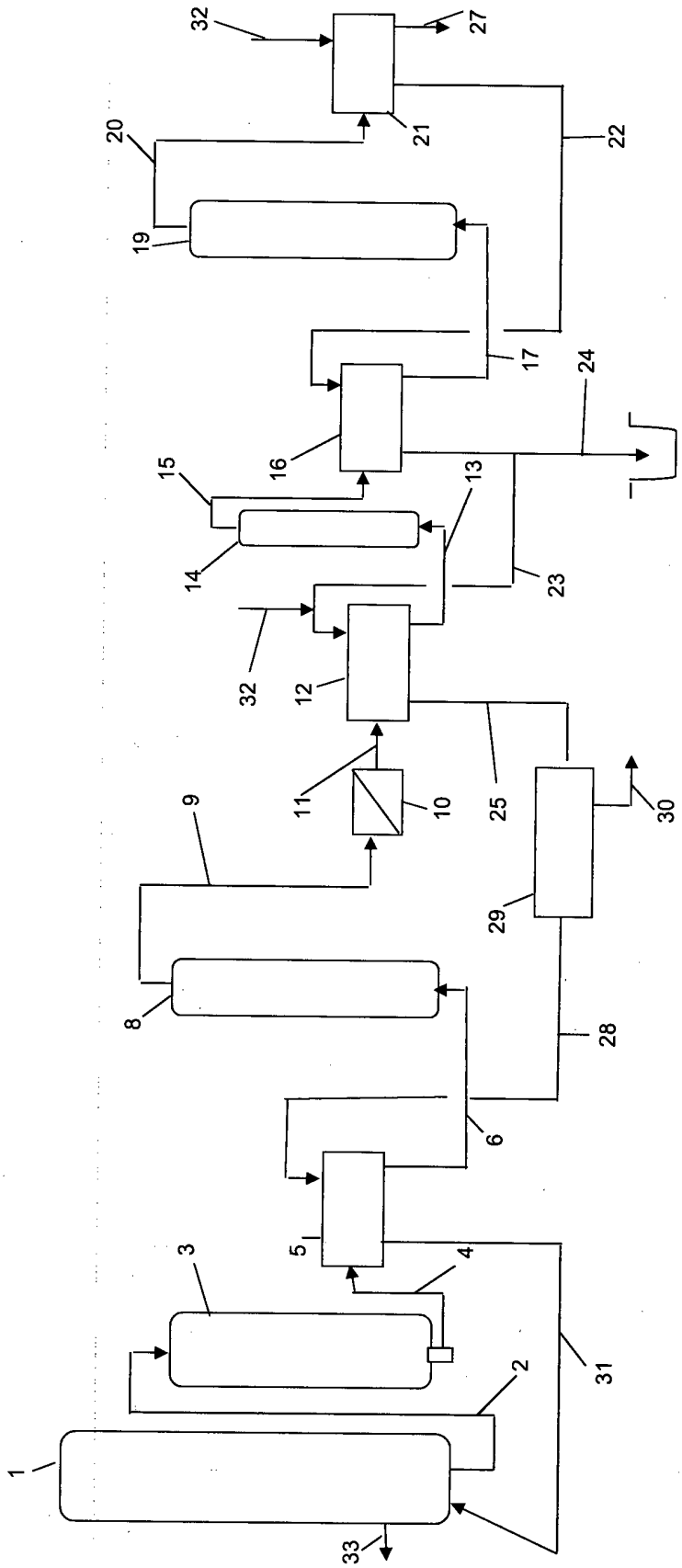

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FIG. 2

REFERENCES CITED IN THE DESCRIPTION

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