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A method and a device for preparing cellulose pulp

Field of the invention

- 5 The invention relates to a method for preparing cellulose pulp according to the preamble of the appended claim 1. The invention also relates to an apparatus for implementing the aforementioned method in accordance with the preamble of the appended claim 15.

10 Background of the invention

The purpose of pulping is to remove, from a lignin-containing raw material, the lignin which binds fibers to each other, and to detach the fibers from each other so that they can be utilized in the manufacture of cellulose and/or paper. When wood is used as the raw material, cooking is preceded by chipping of the raw material, *i.e.* chopping of the wood to the optimal size required by the pulping process. In sulphate cooking, or so-called kraft pulping, the cooking liquor used is strongly alkaline white liquor which consists primarily of sodium hydroxide (NaOH) and sodium sulfide (Na₂S).

In a continuous cooking method gases, primarily air, are removed from both inside the chips and the spaces between the chips before the cooking by conveying hot steam to the chips. Removing gases from the spaces between the chips decreases the amount of gases that disturb the operation of the digester. In addition, when the gases are removed from inside the chips, the chips absorb cooking chemicals better, which improves the yield of the cooking and the quality of the pulp it provides.

30 After the above-described gas removal stage, the chips are brought into contact with the cooking liquor, that is, they are impregnated with the cooking liquor. The purpose of the impregnation is to diffuse the cooking chemicals as evenly as possible into the chips and to impregnate the chip with the cooking liquor. In the impregnation, the cooking liquor penetrates quickly the cavities of the steam-processed chips by the effect of a pressure difference or capillary forces. After the chips

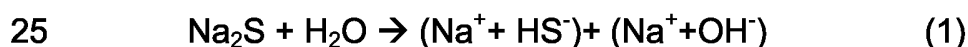
have been impregnated with the cooking liquor, the mass transfers of the active chemicals of the cooking liquor take place, effected by diffusion. The most important factors in the diffusion rate are the concentration of the chemicals in the liquid outside the chip and in the liquid
5 penetrated into the chip, as well as the temperature of the liquid.

After the impregnation, the chips are led to cooking. During the cooking, the dissolution of lignin, or delignification, can be divided roughly into three steps according to the rate of reaction and the selectivity.
10 These steps are initial delignification, bulk delignification and residual delignification. Of these steps, initial delignification takes place, in fact, already in the impregnation stage when the chips are impregnated with the cooking liquor. In the initial step, the rate of reaction of lignin is low, due to the low temperature (typically <140°C). The initial step is characterized by a high alkali consumption which is primarily due to the
15 neutralization of acid groups resulting from reactions of dissolution of hemicellulose, as well as from reactions of extractives. In the initial step, only 15 to 25% of the total quantity of lignin is dissolved. The cooking step which is most important in view of removal of lignin is the
20 bulk step which starts, depending on the raw material, at the temperature of about 135 to 150°C, after which the dissolution rate of lignin increases strongly as the temperature rises further. In the bulk step, 70 to 80% of the lignin is removed. The residual delignification step begins after about 90% of the lignin has been dissolved. Thus, the dissolution
25 rate of lignin is slowed down and is exceeded by the dissolution rate of carbohydrates.

In the sulphate process, factors affecting the cooking include primarily the properties of the wood and the quality of the chips, the cooking time
30 and temperature, as well as the alkali/wood ratio, the liquid/wood ratio and the sulfide content of white liquor. The quality of the chips, particularly their size and dimensions, is a decisive factor in the success and the rate of cooking. In particular, the thickness of a chip affects the distance and rate of penetration of chemicals into the central parts of
35 the chip. Increasing the temperature increases both the transfer rate and the reaction rate. The cooking time and temperature affect the

pulping in such a way that the higher the end temperature of the cook, the shorter the cooking time. The cooking time is also reduced if the temperature is quickly raised to the end temperature at the beginning of the cooking. The rate of raising the temperature to the starting temperature of the bulk delignification step is a critical factor. If the impregnation is insufficient and the rate of raising the temperature is too high, delignification will only take place in the surface layers of the chips, and the alkali concentration will decrease before it reaches the inner parts of the chips. Furthermore, undesired reactions of condensation of lignin will be possible in the areas with an alkali deficiency, and the separation of the fibers will be incomplete. The alkali/wood ratio refers to the alkali content in relation to the dry weight of wood, and it varies typically from 12 to 25%, depending on the raw material and the desired degree of cooking. With relatively higher alkali contents, cooking takes place faster and production is increased, but simultaneously the amount of chemicals to be recirculated becomes higher and the cooking yield decreases.

Sulfide concentration refers to the concentration of sodium sulfide in white liquor (mol/l). Under the conditions of impregnation and cooking, the active sulfide component is the hydrogen sulfide ion HS^- which is formed in the hydrolysis of sodium sulfide according to the following formula:



As disclosed above, another active component of impregnation and cooking is also formed in the hydrolysis of sodium sulfide, namely the hydroxyl ion OH^- . The hydrogen sulfide concentration of white liquor varies to a great extent, ranging typically from 0.2 to 0.9 mol/l, depending on the raw material and on the machinery and equipment in the pulp mill. In the impregnation and cooking stage, the impregnation liquor and the cooking liquor are diluted because of the chip water, a liquid possibly added to control the liquid/wood ratio of the impregnation and cooking, as well as the reaction water. At the beginning of the impregnation stage, the sulfide content of the impregnation liquor

typically ranges from 0.1 to 0.4 mol/l. During the impregnation and the cooking, the hydroxyl and hydrogen sulfide concentrations of the cooking liquor are reduced, because after the cooking, the cooking liquor contains about 10% of the dosed hydroxyl and about 70% of the hydrogen sulfide. An increase in the hydrogen sulfide concentration will increase the rate of delignification, particularly at low temperatures. Consequently, for the pulping, it would be preferable that the hydrogen sulfide concentration of the cooking liquor, particularly inside the chip, was as high as possible already at the early stage of the delignification, that is, in the impregnation stage and not later than at the beginning of the bulk step. A variety of such methods have been presented for increasing the hydrogen sulfide content of the cooking liquor used in the impregnation stage. One method is to bring black liquor from the cooking stage to the impregnation, as disclosed in US patent 5,053,108. However, this method only provides a relative increase in the concentration of hydrogen sulfide ions when compared to the content of hydroxide ions, and the absolute hydrogen sulfide concentration is not necessarily very high compared to white liquor, for example.

Publication WO 03/062524 discloses a method for improving the cooking yield by increasing the dry matter content in the cooking stage, whereby black liquor from cooking is evaporated and recirculated to the beginning of the cooking stage. Black liquor thus formed is led from the end of the cooking stage to the impregnation stage. This method increases the absolute hydrogen sulfide concentration in the cooking stage but fails to provide a real benefit. That is to say, it has been found that the sulfide concentration should be high as early as during the initial delignification, *i.e.* the impregnation, to fully utilize the yield benefit and the increase in the rate of the cooking reactions. In the method described in said publication, the hydrogen sulfide concentration of the initial delignification is reduced, compared to white liquor, by both the washing liquid mixed with the black liquor of the cooking, and the water entering with the chips. Furthermore, the black liquor from the cooking contains a lot of dissolved lignin which, when led to the impregnation, slows down the initial delignification in the impregnation stage and further the bulk delignification in the actual cooking stage,

according to the law of mass action. Also, the reactions of condensation of lignin are increased, particularly at low alkali levels.

5 In SE patent 521678, the sulfide concentration is increased in the impregnation stage by introducing black liquor from pulping to it, the dry matter content of the black liquor being raised by evaporating water from it. By means of this method, the hydrogen sulfide concentration of the cooking liquor used in the impregnation stage and the early pulping steps is increased to some extent. A problem in this method is that the
10 hydrogen sulfide concentration of black liquor from the cooking stage is not very high in practice, because it is reduced by the chip water in the impregnation stage preceding the cooking stage. To obtain a sufficiently high hydrogen sulfide concentration in the black liquor after the cooking for it to be beneficial in the impregnation stage, the system
15 according to the method of the SE patent requires that a lot of white liquor is added into the cooking stage, which has a negative effect on the yield of the pulping, because both a high hydroxyl content and a high temperature occur simultaneously in the bulk step. Also in this application, the black liquor from the cooking slows down the cooking
20 reactions.

Brief description of the invention

25 Consequently, it is an aim of the present invention to provide a method and an apparatus avoiding the problems of the above-presented methods and raising the sulfide concentration of the impregnation liquor used in the impregnation stage as high as possible. As a result, the delignification reactions become faster and the carbohydrate yield of the pulping is improved.

30 To attain this purpose, the method according to the invention is primarily characterized in what will be presented in the characterizing part of the independent claim 1.

The apparatus according to the invention, in turn, is primarily characterized in what will be presented in the characterizing part of the independent claim 15.

- 5 The other, dependent claims will present some preferred embodiments of the invention.

The invention is based on the idea that the hydrogen sulfide concentration of the impregnation liquor used for impregnation is raised by
10 concentrating the impregnation liquor used for impregnation and by recirculating the concentrated impregnation liquor back to the impregnation stage. Evaporation of water will raise the hydrogen sulfide concentration of impregnation liquor high. Also, the hydroxyl concentration of impregnation liquor will remain sufficiently high in spite of the reactions of dissolution of carbohydrates. This will prevent the condensation
15 reactions of lignin. In the concentration of impregnation liquor, free water is evaporated from the impregnation liquor, wherein the concentration differences required by diffusion remain high and the diffusion rate increases. Thanks to the recirculation of the impregnation liquor, the carbohydrates dissolved in connection with the hydrolysis at the
20 beginning of the impregnation are returned to the impregnation, which improves the total yield of the process according to the law of mass action. Furthermore, the carbohydrates thus saved have been found to have a catalyzing effect on the pulping.

25 All of this makes it possible to use a higher impregnation temperature in the method according to the invention than in methods of prior art, namely a temperature ranging from 100 to 170°C, preferably from 100 to 130°C at the beginning of the impregnation and from 120 to 150°C at
30 the end of the impregnation. Thanks to the high temperature of impregnation, the hydrogen sulfide in the impregnation liquor reacts with the lignin and binds chemically to the wood matrix of the chips. As a result, the actual cooking becomes faster and the total cooking yield increases. The high impregnation temperature does not only make the
35 absorption more efficient, wherein no absorption takes place in the cooking stage any longer, but it also provides that the acids formed in

the dissolution of carbohydrates are neutralized already in the impregnation stage and not in the cooking stage, wherein less alkali is needed for digesting the cellulose in the cooking stage, that is, the cooking is more selective.

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In the method according to the invention, a lot of alkali is consumed for reactions of neutralization, extractives and the like in the impregnation stage, and at the same time, impregnation liquor is strongly recirculated; therefore, all or almost all of the white liquor can be added into
10 the impregnation stage without increasing the hydroxyl concentration of the impregnation liquor momentarily too high, which would have a harmful effect on the properties of the cellulose, such as the viscosity or the cooking yield. The white liquor is added to the impregnation liquor before it is concentrated. The addition of the white liquor to the
15 impregnation liquor results in a high concentration of hydrogen sulfide, which is beneficial in the reactions between hydrogen sulfide and lignin in the impregnation stage and at the beginning of the bulk step. Also, the amount of alkali transferred to the cooking stage is significantly reduced, which improves the viscosity of the pulp, because the diges-
20 tion of cellulose begins at high temperatures in the presence of an alkali. This also improves the total cooking yield, because the crystallized cellulose in the wood raw material does not react at the temperatures of the impregnation stage yet, in spite of the presence of the alkali.

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The white liquor to be added into the impregnation liquor before its concentration also reduces bubbling of the impregnation liquor to be evaporated, and makes the dewatering more efficient.

30 The concentration or dewatering of impregnation liquor may be effected by any method suitable for evaporating liquor, for example by expanding heated liquor or evaporating it. Preferably, for evaporating the impregnation liquor, at least one evaporator unit of an evaporation plant integrated in the chemical recovery plant of the pulp mill is used.
35 When the impregnation liquor is dewatered, the content of sodium

sulfide therein rises in relation to the content of sodium hydroxide; in other words, its sulfidity increases.

5 The concentrated impregnation liquor from the evaporator unit is led into a tank where its retention time is adjusted to be such that potash soap, or the like, in the impregnation liquor rises on the impregnation liquor in the tank, and can be separated from it. In this way it is possible to reduce the COD load in the cooking and washing stages following the impregnation stage. The tank used for soap separation is preferably a tank existing in the evaporation plant.

By means of the invention, the hydrogen sulfide concentration of the impregnation liquor can be doubled compared to the process of prior art which uses black liquor for impregnation.

15 The cooking yield can be improved further by adding polysulfides into the evaporated impregnation liquor. Polysulfides oxydate the reactive terminal groups of the carbohydrates to be alkali stable. The polysulfides can be formed, for example, in the pulping apparatus by adding black liquor obtained from the cooking and containing added oxygen, to the concentrated impregnation liquor. The oxygen oxydates the lignin structures of the black liquor to quinone compounds which reduce hydrogen sulfide in impregnation liquor into polysulfides.

25 When the chips are transferred from the impregnation stage to the cooking stage, impregnation liquor is transferred to the cooking stage primarily within the chips and to a small extent in the form of so-called free liquid outside the chips, the quantity of the liquid being kept as small as possible by arrangements of process engineering. The liquid/wood ratio in the cooking stage can be adjusted as desired by recirculating liquor obtained from cooking to the beginning of the cooking, but not to the impregnation stage.

35 By means of the method according to the invention, conditions of impregnation are achieved, in which the concentrations of dissolved organic and inorganic dry matter in the impregnation liquor are high

when compared with methods of prior art. By the apparatus of the invention, it is possible to separate the liquid circulations in the impregnation and cooking stages from each other and to utilize the existing machinery and equipment in the pulp mill.

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In addition to the numerous advantages mentioned above, the invention has the advantage that the hydrogen sulfide concentration of the impregnation liquor to be used in the impregnation stage can be raised as high as possible, compared to the hydrogen sulfide concentration of white liquor, wherein the yield from the cooking stage following the impregnation stage is improved. Furthermore, thanks to the reactions between lignin and sulfide in the impregnation stage, the bulk step of pulping becomes faster. Moreover, less water is conveyed with the impregnated chips into the cooking stage, which makes the cooking more efficient. Because the quantity of solution to be heated in the cooking stage is smaller, it is possible to reduce the cooking temperature, and the quantity of high-pressure primary steam used in the cooking stage becomes smaller. Furthermore, because the temperature at the impregnation stage is provided by secondary energy obtained from the cooking, the high-steam vapour saved from the cooking can be transferred to a turbine, to produce more electricity than before. The invention thus improves the energy economy and profitability of the whole mill.

25 Brief description of the drawings

In the following, the invention will be described in more detail with reference to the appended Fig. 1 which shows, in a schematic view, a cellulose pulping device according to the invention.

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Detailed description of the invention

In this description, the term impregnation liquor refers to the alkaline solution used in the impregnation stage, consisting primarily of white liquor. The effective agents in white liquor are sodium hydroxide

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(NaOH) and sodium sulfide (Na_2S). The term line refers to any tube, duct or channel suitable for conveying a liquid, a gas, or a suspension.

Figure 1 is a schematic view showing a device for cellulose pulping according to the invention. The chips used as raw material for cellulose pulp are first subjected to a gas removal stage. In the gas removal stage, the chips are treated in a gas removal unit 2 with a hot gaseous medium, such as steam, to remove gases from within and between the chips. After the gas removal, the chips are transferred by a transfer device 3 via a line 4 to the impregnation stage. Impregnation liquor from an impregnation liquor tank 6 is also supplied via a line 7 into the line 4.

In the embodiment of Fig. 1, the impregnation stage is shown in two steps, which is typical particularly for continuous pulping processes. The impregnation is effected in the impregnation stage, in an impregnation vessel 5, in which the first step 5a and the second step 5b of the impregnation are typically connected to each other, for example placed on top of each other in the same vessel in such a way that the flow of chips is directed downwards through the steps. The chips are brought to the impregnation from the upper end of the first impregnation stage 5a, together with the impregnation liquor, and passed through this step and further down to the second impregnation stage 5b. In the first impregnation stage 5a, the impregnation is effected downstream, and during it, the concentration of hydrogen sulfide in the impregnation liquor inside the chip increases because the hydroxide in the impregnation liquor is quickly consumed at the beginning of the impregnation. The water in the chips is diffused out of the chips into the impregnation liquor, diluting it. This diluted impregnation liquor is led from the end of the first impregnation stage 5a to be concentrated along the line 26. The first impregnation stage takes 15 to 120 min, preferably 15 to 45 min.

In the second impregnation stage 5b, the chips are impregnated upstream with the impregnation liquor introduced from the impregnation liquor tank 6 via a line 8. The impregnation liquor is heated in a

heat exchanger 9 before it is led to the second impregnation stage 5b. The heating of the impregnation liquor increases the temperature of the second impregnation stage 5b, wherein the reactions between sulfide and lignin become faster. If necessary, the second impregnation stage 5b can also be arranged downstream. One aim of the second step is to increase the alkali concentration inside the chips so that the alkali content inside the chip is also sufficient for the cooking stage. Another aim of the second impregnation stage is to displace the rest of the water in the chips and have it to be concentrated together with the impregnation liquor obtained from the second stage 5b. The water-containing impregnation liquor is led from the second stage to be concentrated along the line 26. The second impregnation stage 5b takes 5 to 60 min, preferably 10 to 45 min. The impregnation stage may also consist of a single stage only, wherein the chips are impregnated downstream in the impregnation vessel. In the impregnation stage 5, the temperature ranges from 100 to 170°C. Preferably, the temperature is lower in the first impregnation stage 5a than in the second impregnation stage 5b. The temperature ranges preferably from 100 to 135°C in the first impregnation stage 5a and preferably from 120 to 150°C in the second impregnation stage 5b.

White liquor is led via line 31 to the line 26 which thus conveys impregnation liquor to be concentrated from the first impregnation stage 5a and the second impregnation stage 5b. The addition of white liquor is necessary, not only to prevent bubbling of the impregnation liquor to be evaporated and to make dewatering more efficient, but also to achieve the alkali content required for cooking.

As already described above, the impregnation stage comprises the impregnation of the chips with the cooking solution, and the initial delignification. From the impregnation stage, the chips are led together with a part of the impregnation liquor via a line 10 to the cooking stage for the actual bulk delignification and, if desired, also the residual delignification, that is, the actual cooking. The cooking takes place in a pulping vessel 11 at a temperature from 130 to 190°C, depending on the wood raw material, and it takes 30 to 150 min. The cooking stage

may comprise both a downstream and an upstream portion whose alkali concentrations and temperatures can be controlled. The fibers detached or separated from each other during pulping are transferred via a line 12 to be washed in a washing vessel 13, *i.e.* to a washing stage. The purpose of washing is to separate that part of lignin which comes with the fibers, and the cooking liquor from the fibers. The washed pulp is led to further processing via a line 14.

The above-mentioned stages of impregnation and cooking can be performed either one by one in separate vessels, or together in one and the same vessel, one by one. The stages can also be carried out in an elongated vessel on top of each other so that the impregnation stage is at the top. The chips thus move downwards from one step to another in the vessel.

The lignin-containing liquor, or black liquor, used in the pulping and obtained from the cooking stage, is led to the chemical recovery plant of the pulp mill. The purpose of the chemical recovery is to regenerate the chemicals used in the pulping to such a form that they can be re-used in the pulping. In practice, this means that the black liquor obtained from the cooking and the washing fluids obtained from the washing of the pulp are led to be burned in a recovery boiler, in which a sodium carbonate containing smelt is obtained as a result of combustion. The smelt is dissolved and causticized, resulting in white liquor which can be led to be used as impregnating and cooking liquor.

Before the black liquor and the washing fluids obtained from the washing of the pulp are led to be burned in the recovery boiler, they are dewatered. The dewatering or concentration of black liquor is effected by means of an evaporation plant provided in the chemical recovery plant. The evaporation plant normally consists of several evaporator units in which black liquor is heated indirectly under pressure. As a result of the heating, the liquor swells and water is separated from it. The evaporator units are connected to each other in such a way that the liquor passes in them from one unit to another, wherein in each step, the dry matter content of the liquor to be concentrated is higher

than the liquor concentrated in the preceding step. The structure of the evaporator units and the principles of their operation as well as the principles of operation of the evaporation plant are obvious for a person skilled in the art, and they will therefore not be described in more detail in this context.

In the embodiment of the invention shown in Fig. 1, the black liquor and pulp washing fluids obtained from the cooking stage and the pulp washing stage are led to a weak liquor tank 15 in connection with the chemical recovery plant. The flow of black liquor is indicated with the letter C, and the flow of washing fluid obtained from the pulp washing is indicated with the letter D. The black liquor, which thus also contains pulp washing fluid, is led from the tank 15 along a line 16 to an evaporation plant 17. In this embodiment, the evaporation plant consists of five evaporator units 17a to 17e. The black liquor is led to the last unit 17e of the evaporation plant, in which it is dewatered, and the black liquor thus concentrated is led via a line 18 to the next evaporator unit 17d. From the evaporator unit 17d, the liquor is led via a line 19 to the evaporator unit 17b and further via a line 20 to the last evaporator unit 17a. From the last evaporator unit 17a, the liquor is led along a line 21 to be burned in a recovery boiler (not shown in the figure). In each evaporator unit, the black liquor is dewatered, wherein its concentration is always higher in the next step than in the preceding step. Also, the operation of each evaporator unit substantially includes an internal circulation in which the liquor to be evaporated is recirculated by a pump 22 onto the evaporating surfaces of the evaporator unit. The heating steam supplied to the evaporator units, shown by a dotted line in the figure and being, for example, low-pressure secondary steam from the pulp mill, is led via a line 23 to the last evaporator unit 17a of the liquor circulation. The heating steam is brought to contact with tubular or lamellar evaporating surfaces arranged inside the evaporator unit for indirect heating of black liquor. The condensate of the heating steam is discharged via an outlet (not shown in the figure) in the evaporator units. The hot secondary steam separated from each evaporator unit by evaporation of the liquor is led as heating steam along a duct 24 for the next step. The secondary steam obtained from

- the first evaporator unit 17e is led via a line 25 either to the combustion of non-condensable gases of the pulp mill, or to be stripped. In the embodiment of Fig. 1, the operation of the evaporator units and the transfer of heating steam from one step to another is shown in a significantly reduced manner. Naturally, it is also possible to supply fresh heating steam to other evaporator units than the last evaporator unit 17a. Also other arrangements for recirculating and transferring the heating steam are possible.
- 10 As disclosed above, according to the invention not all evaporator units in the evaporation plant are used for evaporating the black liquor, but one evaporator unit in the same heating medium circulation is by-passed. According to the invention, this evaporator unit is used for evaporating the circulating impregnation liquor obtained from the
- 15 impregnation vessel 5, from the impregnation stages 5a and 5b, and the circulating liquor obtained from the transfer device. In other words, the liquor circulation of the black liquor circulating in the evaporator units is separated from said evaporator unit. The impregnation liquor obtained from the first stage 5a and the second stage 5b of the impregnation stage and the circulating liquor from the transfer device 3 are led
- 20 along a line 31 to be concentrated in the third evaporator unit 17c of the evaporation plant. The impregnation liquor evaporated from the evaporator unit 17c is led along a line 27 to the impregnation liquor tank 6, from which the impregnation liquor is recirculated to the first
- 25 impregnation stage 5a and the second impregnation stage 5b via the lines 7 and 8. In this way, a circulation of impregnation liquor is provided. Also, the circulating liquor required by the transfer device 3 is returned via the line 7.
- 30 By evaporating free water from the impregnation liquor, it is possible to maintain the high concentration differences required for diffusion in the impregnation. The evaporator unit 17c used for evaporating the impregnation liquor can be, in principle, any evaporator unit of the evaporation plant. It is advantageous to use such a unit which yields,
- 35 as a result of evaporation, impregnation liquor with a dry matter content at the same level as that of the impregnation liquor. The evaporator

unit 17c used for evaporating the impregnation liquor is in the same heating medium circulation with the other evaporator units 17a, 17b, 17d, 17e of the evaporation plant. The expansion steam obtained as a result of evaporating the impregnation liquor is thus led as heating steam via the line 24 to the next evaporator unit 17d.

By means of the invention, the hydrogen sulfide concentration of the impregnation liquor can be raised higher, which improves, as already said above, the cooking yield. When one or more evaporator units of the evaporation plant already existing in the pulp mill are used for concentrating the impregnation liquor, no extra investments in machinery and equipment will be needed for implementing the invention. The impregnation liquor tank 6 is preferably a tank existing in the evaporation plant, for example a feed liquor tank. These are normally equipped with an apparatus for separating soap. The introduction of the invention will not require a long shutdown for construction, but it can be easily taken into use during a normal maintenance shutdown, as it only requires some pipeline construction.

To make the removal of soap from the impregnation liquor possible, the retention time of the evaporated impregnation liquor in the impregnation liquor tank 6 is adjusted to be such that the soap therein accumulates on the impregnation liquor and can be discharged via a line 28.

The cooking yield can be improved further by adding polysulfides to the evaporated impregnation liquor. According to one embodiment of the invention, black liquor B obtained from the cooking stage is led via a line 29 indicated with broken lines to the line 27 conveying evaporated impregnation liquor. Oxygen is led via a line 30 into the black liquor, in the line 29, before the addition of the black liquor into the impregnation liquor line 27. The oxygen oxydates the lignin structures of the black liquor into quinone compounds. In the impregnation liquor tank 6, the quinone compounds reduce hydrogen sulfide in the impregnation liquor into polysulfides.

The invention also makes it possible to use anthraquinone, which is expensive as such, to increase the yield in the manufacture of sulphate cellulose pulp. Thanks to the invention, the dosage of anthraquinone can be reduced, because the concentration of the aqueous impregna-
5 tion liquor concentrates all the chemicals therein.

Naturally, the above-presented apparatus for preparing a fiber pulp also comprises a large number of process devices, such as sieves, pumps and valves, whose placement and operation in the pulping
10 apparatus are obvious as such for a person skilled in the art and which are, for clarity, not shown in the figure.

The invention is not intended to be limited to the embodiments presented as examples above, but the invention is intended to be applied
15 widely within the scope of the inventive idea as defined in the appended claims. The invention can thus be applied in various types of pulping methods and digesters, such as a continuous pulping method and its various modifications, or batch digesting, particularly displacement batch digesting. The raw material used can be any lignocellulose
20 material, such as softwood, hardwood, bagasse or eucalyptus.

Furthermore, the evaporation plant used as a part of the invention is not tied in any way to the above-presented embodiment. Consequently, the number of evaporator units arranged in the evaporation plant may
25 be different from that presented above. Also, the invention is not limited to any given evaporator model, any number, shape or placement of evaporating surfaces in the evaporator units, or the circulation of heating medium inside the evaporator. It is also possible to use more than
30 one evaporating units for evaporating the impregnation liquor.

Claims

1. A method for preparing cellulose pulp, the method comprising at least
- 5 - an impregnation stage, in which a chipped lignin-containing raw material is impregnated with an alkaline impregnation liquor, and
- a cooking stage, in which the lignin-containing raw material, impregnated with impregnation liquor, is cooked, and
- 10 in which method the black liquor obtained from the cooking stage is concentrated in an evaporation plant (17) integrated in a chemical recovery plant,
- characterized** in that
- in the impregnation stage, the lignin-containing raw material is
- 15 impregnated with a circulating alkaline impregnation liquor, which impregnation liquor is concentrated by evaporating water from it in at least one evaporator unit (17c) of the evaporation plant (17) arranged in the chemical recovery plant of a pulp mill.
- 20 2. The method according to claim 1, **characterized** in that before the concentration, white liquor is added to the impregnation liquor.
3. The method according to claim 1, **characterized** in that impregnation liquor is led from the impregnation stage to at least one evaporator
- 25 unit (17c) of the evaporation plant (17) arranged in the chemical recovery plant of the pulp mill, and is concentrated therein.
4. The method according to claim 1, **characterized** in that the concentrated impregnating liquor is led from the evaporator unit (17c) into
- 30 an impregnation liquor tank (6) which is an existing tank in the evaporation plant (17), such as a feed liquor tank.
5. The method according to claim 4, **characterized** in removing the soap accumulated on the concentrated impregnation liquor in the
- 35 impregnation liquor tank (6).

6. The method according to claim 1, **characterized** in that the impregnation stage comprises a first impregnation stage (5a) and a second impregnation stage (5b).
- 5 7. The method according to claim 4 or 6, **characterized** in that the concentrated impregnation liquor is led from the impregnation liquor tank (6) to the first impregnation stage (5a) and the second impregnation stage (5b).
- 10 8. The method according to claim 7, **characterized** in that the concentrated impregnation liquor is heated before it is led into the second impregnation stage (5b).
- 15 9. The method according to claim 6, **characterized** in that the lignin-containing raw material is impregnated downstream in the first impregnation stage (5a) and upstream in the second impregnation stage (5b).
- 20 10. The method according to claim 6 or 9, **characterized** in that the impregnation temperature is 100 to 170°C and that the first impregnation stage (5a) takes about 15 to 120 min, preferably about 15 to 45 min, and the second impregnation stage (5b) takes about 5 to 60 min, preferably about 10 to 45 min.
- 25 11. The method according to claim 1, **characterized** in that the impregnation liquor obtained from the impregnation stage is concentrated in at least one evaporator unit (17c) of the evaporation plant (17), the evaporator unit (17c) being in the same heating medium circulation as the other evaporator units (17a, 17b, 17d, 17e) of the evaporation plant (17).
- 30 12. The method according to claim 1, **characterized** in that the impregnation liquor obtained from the impregnation stage is concentrated in at least one evaporator unit (17c) of the evaporation plant (17), the evaporator unit (17c) being separated from the liquor circulation of black liquor.
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13. The method according to claim 11, **characterized** in that the secondary steam released by the concentration of impregnation liquor is led as heating medium to the evaporation plant (17).

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14. The method according to claim 1, **characterized** in that the method also comprises

- a gas removal stage, in which the chipped lignin-containing raw material is treated with a steam for gas removal, and
- 10 - a washing stage, in which the cellulose fibers released by cooking are washed.

15. An apparatus for preparing cellulose pulp, the apparatus comprising

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- impregnating means (5) for impregnating a chopped lignin-containing raw material with alkaline impregnation liquor, and
- cooking means (11) for cooking the lignin-containing raw material impregnated with impregnation liquor, and

the apparatus further comprising an evaporation plant (17) integrated in a chemical recovery plant, arranged for concentration of black liquor obtained from the cooking of the lignin-containing raw material,

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characterized in that

- at least one evaporator unit (17c) in the evaporation plant (17) forming a part of the chemical recovery plant of a pulp mill is arranged for concentrating impregnation liquor, which concentrated impregnation liquor is arranged to be used for impregnating a lignin-containing raw material.

25

30 16. The apparatus according to claim 15, **characterized** in that the lignin-containing raw material is arranged to be impregnated with a circulating alkaline impregnation liquor.

17. The apparatus according to claim 15, **characterized** in that before the concentration, white liquor is arranged to be added to the impregnating liquor.

35

18. The apparatus according to claim 15, **characterized** in that impregnation liquor, obtained from the impregnation of the lignin-containing raw material, is arranged to be led to at least one evaporator unit (17c) of an evaporation plant (17) arranged in the chemical recovery plant of the pulp mill, to be concentrated therein.

19. The apparatus according to claim 15, **characterized** in that the an impregnation liquor tank (6), into which the concentrated impregnating liquor is arranged to be led from the evaporator unit (17c), is an existing tank in the evaporation plant (17), such as a feed liquor tank.

20. The apparatus according to claim 19, **characterized** in that the impregnation liquor tank (6) is connected to a line (28), through which soap accumulated on the concentrated impregnation liquor in the impregnation liquor tank (6) is arranged to be discharged.

21. The apparatus according to claim 15, **characterized** in that the impregnating means (5) constitute an impregnation stage which is divided into a first impregnation stage (5a) and a second impregnation stage (5b), and that the lignin-containing raw material is arranged to be impregnated in the first impregnation stage (5a) and the second impregnation stage (5b).

22. The apparatus according to claim 19 or 21, **characterized** in that the concentrated impregnation liquor is arranged to be led from the impregnation liquor tank (6) to the first impregnation stage (5a) and the second impregnation stage (5b).

23. The apparatus according to claim 22, **characterized** in that the apparatus comprises a heat exchanger (9) for heating the concentrated impregnation liquor before it is led into the second impregnation stage (5b).

24. The apparatus according to claim 21, **characterized** in that the lignin-containing raw material is arranged to be impregnated

downstream in the first impregnation stage (5a) and upstream in the second impregnation stage (5b).

25. The apparatus according to claim 21 or 24, **characterized** in that
5 the impregnation temperature is 100 to 170°C and that the first
impregnation stage (5a) takes about 15 to 120 min, preferably about 15
to 45 min, and the second impregnation stage (5b) takes about 5 to
60 min, preferably about 10 to 45 min.
- 10 26. The apparatus according to claim 15, **characterized** in that at least
one evaporator unit (17c) of the evaporation plant (17), in which the
impregnation liquor obtained from the impregnating means (5) is
arranged to be concentrated, is in the same heating medium circulation
15 as the other evaporator units (17a, 17b, 17d, 17e) of the evaporation
plant (17).
27. The apparatus according to claim 15, **characterized** in that at least
one evaporator unit (17c) of the evaporation plant (17), in which the
impregnation liquor obtained from the impregnating means (5) is
20 arranged to be concentrated, is separated from the liquid circulation of
black liquor.
28. The apparatus according to claim 26, **characterized** in that the
expansion steam released by the concentration of impregnation liquor
25 is arranged to be led as heating medium into the evaporation plant
(17).
29. The apparatus according to claim 15, **characterized** in that the
apparatus also comprises
- 30 - gas removal means (2) for removing gases from the chipped
lignin-containing raw material, and
- washing means (13) for washing the cellulose fibers released by
cooking.

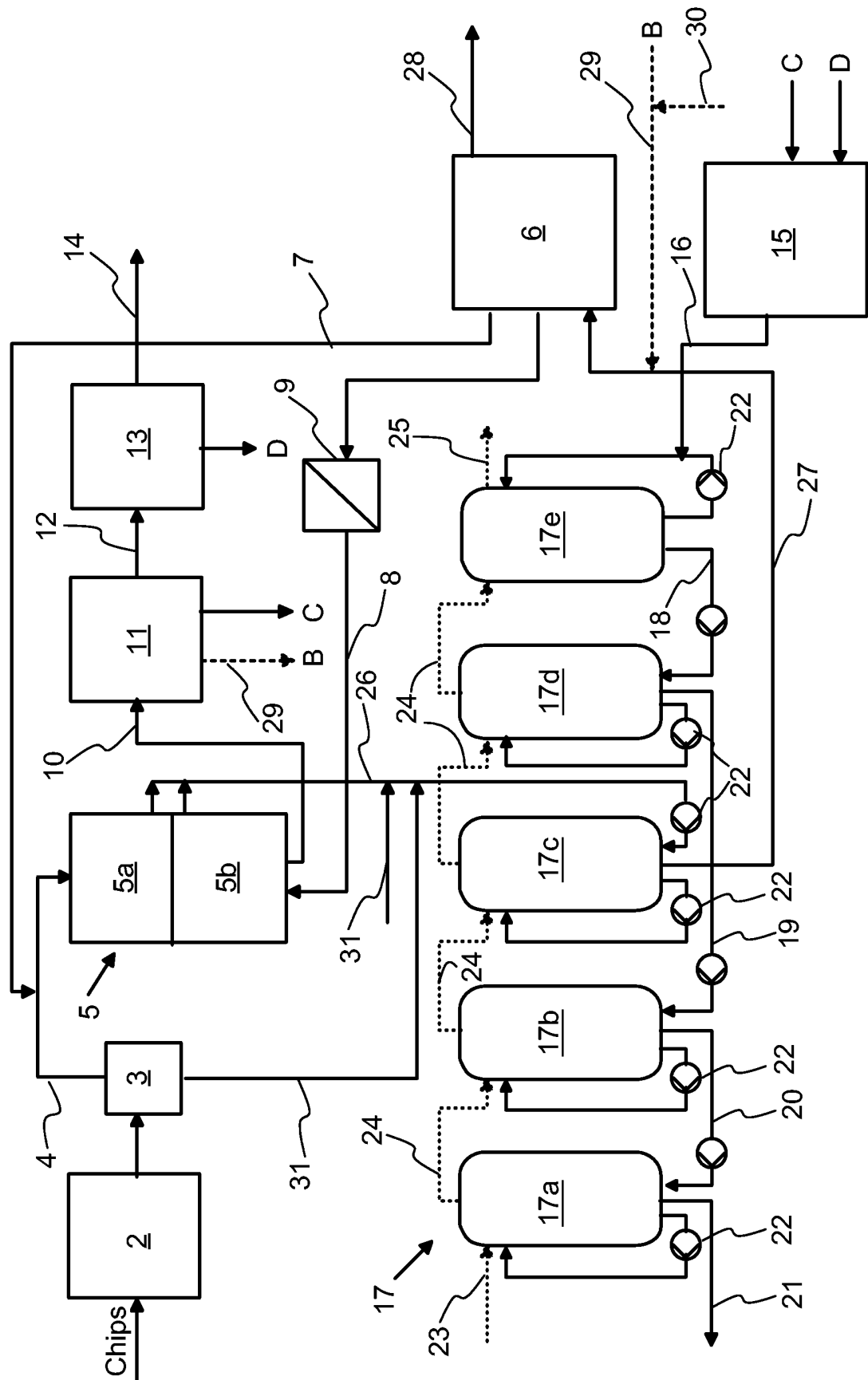


Fig. 1

INTERNATIONAL SEARCH REPORT

International application No

PCT/FI2005/050343

A. CLASSIFICATION OF SUBJECT MATTER
D21C1/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
D21C

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

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

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	WO 03/062524 A (KVAERNER PULPING AB; LINDSTROEM, MIKAEL; SNEKKENES, VIDAR) 31 July 2003 (2003-07-31) cited in the application claims 1-10; figure 1 -----	1, 15
X	US 5 674 359 A (CHASSE ET AL) 7 October 1997 (1997-10-07) claims 1-24; figure 2 -----	1, 15
A	EP 0 903 436 A (KVAERNER PULPING AB) 24 March 1999 (1999-03-24) the whole document -----	1-29
A	US 5 080 755 A (BACKLUND ET AL) 14 January 1992 (1992-01-14) the whole document -----	1-29
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☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

International application No
PCT/FI2005/050343

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
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A	US 3 258 390 A (II GEORGE H. TOMLINSON) 28 June 1966 (1966-06-28) the whole document -----	1-29

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Information on patent family members

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