A continuous, alkaline process for the production of pulp from wood chips, wherein the preheated chips are subjected to an extended impregnation step for at least 60 min, preferably longer, at a temperature not exceeding the impregnation liquor boiling point at atmospheric conditions, and a rapid heating and cooking period of less than 65 min, preferably shorter, followed by cooling to below reaction temperature. Fresh alkali is added both during impregnation and the heating/cooking period.
Figure 1

Conventional Batch

Temperature

Time
Figure 3

Continuous Cooking

Temperature

Time
METHOD FOR ALKALINE COOKING OF FIBER MATERIAL

FIELD OF THE INVENTION

[0001] The invention relates to the field of alkaline pulp- ing.

BACKGROUND OF THE INVENTION

[0002] Among the chemical pulping methods, alkaline cooking processes and especially kraft cooking are dominant in the production of cellulose or chemical pulp because alkaline cooking provides pulp fibers which are stronger than those from any other commercial pulping process. The lignocellulosic material, typically chopped into wood chips, is treated in either batch or continuous digesters.

[0003] In chemical pulping processes as alkaline cooking, the chemical reactions with wood components are heterogeneous-phase border reactions. To ensure uniform reactions, it is vital that all fibers in the wood get their proper share of chemicals and energy. Non-uniformity occurs when these criteria are not fulfilled and this may result in a larger amount of unfibered wood (rejects) in the final pulp, discolored pulp, lower final yield and impaired bleachability and paper properties. The objective of chemical pulping is to remove lignin so that the fibers can separate. Ideally, each fiber should receive the same amount of chemical treatment for the same time at the same reaction site. This means that chemicals and energy must be transported uniformly to reaction sites throughout each chip. There are two major stages where this can happen; (1) the impregnation phase where chips are saturated with chemical-containing liquid before delignification reactions begin and (2) the continuous movement of chemicals to the reaction sites during the cooking phase. Chip dimensions are of major importance in this context. The longer, wider and especially the thicker the chips are, the longer is the transportation distance to the centers of the chips.

[0004] Pores inside fresh wood chips are also partly filled with liquid and partly with air. The ratio is, among others, determined by the moisture content or dry content of wood. The air should be removed from the chips before they can be fully impregnated by cooking liquor. This is usually done by pre-steaming the chips.

[0005] As cooking proceeds, reactive ions must diffuse into the chips. If the transportation distance is too long and rate of transportation too slow, the chemicals may be completely consumed before they can reach the chip centers, resulting in non-uniform cooking. Thus, in cooking there is a critical balance between the rate of ion transportation, wood porosity, chip dimensions and the rate of chemical reaction. E.g. raising the temperature increases the rate of transportation, but the rate of reaction increases still faster. On an average, long and thick chips will not delignify as fast as short and thin chips when the same cook parameters are used. In conventional batch cooks, long and thick chips generate more screening rejects than short and thin chips and shorter cooks and higher cooking temperatures aggravate the effect. The higher the cooking temperature, the shorter is the required time to a certain delignification degree and the higher is the rate difference between delignification reaction and ion transportation. Thus it seems that cooking uniformity would require that chips should be small and thin enough and that the size variation in the chip furnish should be as narrow as possible. However, in practice many other important parameters as e.g. chipping parameters, chipping capacity, fiber cutting, fiber length, paper properties, the bulk density of the chip column, the permeability of the chip column, saw dust and pin chips amounts and plugging of digester screens has also to be taken into account. Saw dust is material passing through a 3 mm hole screen; pin chips is material that passes through 7 mm hole screens but is retained on a 3 mm hole screen.

[0006] It has also been proposed that the rate of heating influence the result. A shorter heating period requires a smaller chip size to ensure sufficient uniformity, and it has been demonstrated that shredding of the chips will reduce the screening when using very rapid cooks (cycle-to-cycle) at high temperatures, and almost no heating period. Such conditions, i.e. fast heat-up and high temperatures prevail in certain types of continuous digesters as saw dust and pin chips digesters. In the Kamyr type of continuous digester as well as in most batch digesters, slow cooking, i.e. low rate of reaction, long heating period and lower maximum temperature, allow a more uniform cooking also of ordinary mill size chips. As both shredding and chipping to smaller size will affect fiber length, pulp quality and efficiency of operation, it is desirable to provide conditions that allow the normal industrial chip size.

[0007] For processing ordinary mill size chips, it has been proposed that cooking non-uniformity can be reduced and perhaps eliminated by proper chip manufacturing and screening, proper impregnation and sufficiently low cooking temperature. Thus low rate of chemical reactions and long cooking stage in combination with impregnation and uniform chips.

[0008] In a conventional alkaline batch cooking process, wood chips are fed to a digester and cooking liquor is added. The chips can be steam treated before, during or after chip filling to pre-heat the chips and remove air. The pressure is about atmospheric after liquor addition. When chips and liquor have been added, the cook is immediately started by introduction of heat either indirectly or directly by steam. Impregnation occurs during heating. The cook itself consists of a heating period and an “at pressure” period. Typical heat-up times and at pressure times are 60 to 150 minutes and 60 to 120 minutes, respectively. Typical sum of heat-up and at pressure times is about 150 minutes. At the conclusion of the cook when the delignification has proceeded to the desired reaction degree, a blow valve in the digester is opened and the contents of the digester are discharged into a blow tank as the hot liquor in the digester flashing into steam and forces the cooked pulp out of the digester. The cooked material is cooled and defiberized.

[0009] The difficulty with conventional batch cooking is the non-uniform and poor quality of the pulp, high energy consumption and environmental concerns. Therefore, one of the most important objectives has been the attempt to improve the efficiency of the cooking process and to improve the properties of the resulting pulp and the uniformity, especially in relation to above mentioned conventional process. Continuous flow processes and displacement batch processes have therefore been developed.

[0010] Displacement batch pulping processes were developed in the 80’s, originally for the sake of energy economy.
Following a batch cook, the black liquor was recovered, divided into fractions according to temperature, stored and introduced into a digester charged with fresh lignocellulosic material in order to transfer the heat of the completed cook to a subsequent cook. The total duration of the black liquor impregnation stage in batch displacement processes is typically below 30 min at temperatures below 100 °C. Heating is carried out by displacement with a black liquor having a higher temperature than the impregnation liquor. Following these initial stages, white liquor is introduced, and a main cooking stage follows. Typically, the total duration of the hot liquor fill stage, temperature adjustment and the cooking stage is in the range 95-120 min.

Several variations of the batch displacement cooking process have been developed to optimize utilization of cooking chemicals and to handle variations in raw material, where it may be advantageous to adjust the composition and/or sequence of pretreatment liquors. In addition to energy economy, adjustment possibility, pulp quality and flexibility are advantages when displacement batch cooking processes are used. On the other hand, these processes require a significant amount of tanks and piping to handle the various liquors involved. As the trend is towards closed circuits and lower emissions from plants, accumulation problems may also occur.

In contemporary continuous processes, typically referred to as the Kamyr type, energy savings are achieved by pre-heating the chips with steam obtained from flashing the hot black liquor. In the pre-steaming of the chips, chips are preheated and air is removed from the chips to facilitate later liquor impregnation. In continuous cooking, the chip impregnation zone typically involves 30-60 min or shorter chip retention at a temperature of 115-130°C and a high pressure to enhance the pre-impregnation of the chips and the ion transportation into the chips. Since penetration rates increase with increased pressure, impregnation stages operate at pressures that greatly exceed the liquor saturation pressure at the specified temperature, i.e. typically greater than 10 bars operating pressure for impregnation temperatures of 115-130°C. Subsequent to impregnation, the chips are heated directly in a vapor phase and/or in several liquor heating circuits to full cooking temperature, and then typically cooked for at least 1.5-2.5 hours in a concurrent cooking zone at temperatures below 165°C. Practical experience suggests that the process becomes chemical transportation rate limited at cooking times below about 1.5-2.5 hours and temperatures above 165°C. Therefore, typical cooking temperatures are between 150-165°C, but even cooking temperatures of 140-150°C can also occur, see for example international patent application WO 98/35091. Thus, a minimum of 1.5-2.5 hours of cooking is required. In addition, subsequent to the concurrent cooking zone, a countercurrent zone, typically referred to as the hi-heat zone, usually follows for 24 hours at temperatures of 130-160°C. Contemporary continuous cooking as e.g. ITC, EMCC and Lo-solids cooking retains the cooking temperature, typically 150-160°C, all through the countercurrent zone, i.e. enlarging the cooking zone to the counter-current zone. These modern digesters have thus a total cooking zone of about 240-360 minutes. For the countercurrent zone, washing filtrate is pumped into the bottom of the vessel. The vessel bottom is also a blow dilution and cooling zone. Discharge temperature is typically 85-90°C.

The continuous processes offer, compared to conventional batch digesters: more space efficiency, lower installed power, lower volumes of inlet streams and outlet stream, steady-state operation vs. batch fill and discharge cycles, energy efficiency, lower environmental impact and a first stage of brownstock washing.

However, it is found that while the typical continuous processes have the aforesaid advantages the pulp obtained has a number of properties, e.g. strength and uniformity of the pulp, which are inferior to e.g. pulp produced under well-controlled laboratory conditions. The continuous process still lacks sufficient impregnation and this has to be compensated by lower reaction temperatures and long retention times in the digester. This leads to expensive, large-size and huge digesters designed for high pressures and temperatures. The scale of contemporary digesters, typically of the type Kamyr, at higher production levels also causes mass-transfer problems in liquid circulation and displacement, which further is compensated by longer retention times and lower cooking temperatures.

Impregnation theoretically requires small chips, but modern continuous digesters are based on the principle of maintaining sufficient liquid circulation and a good displacement efficiency. This calls for chip properties that are in conflict with some of the basic requirements for ensuring uniform delignification. Thus, a large chip size must be used, which leads to inferior impregnation and further longer retention times in cooking and expensive technology. Thus, the pulp maker has been trapped by his own technology. It is stated that the 30-60 min retention time at 115-130°C in impregnation zones of continuous mill digesters could never provide a completely uniform distribution of cooking chemical for all chips (mill chips) before the start of bulk delignification.

In Swedish patent application 9602016-9, it is suggested that the way the chips are treated before continuous kraft cooking is disadvantageous for the strength of the pulp. It is proposed that the pre-impregnation at 110-130°C in e.g. Kamyr continuous digesters is unfavorable and the chips should instead be cold impregnated. During impregnation it would only be necessary to have enough alkali present to neutralize possible by-products, in order to prevent formation of acidic regions that can damage the fiber properties. The alkali required during cooking can consequently be added after the pretreatment, and/or during cooking, i.e. a high alkali level is not required during impregnation. A pulping process is disclosed which comprises so-called "cold impregnation" as its main feature. A temperature of about 80-110°C is specified, the time period being unlimited. However, an optimum of 2-3 hours is suggested. Pressure may be used in order to compress gas bubbles and cause sinking of the chips. The theory behind the cold impregnation stage is, that acid-generating processes within the chips shall be suppressed until the chips are filled with alkali sufficient to neutralize any acid released when reaction commences at higher temperatures, and the impregnation step is defined as resulting in "an alkali concentration sufficient to neutralize all acid produced".

The proposed process preferably uses a conventional continuous digester like MCC, EMCC or Lo-solids digester. Thus, the retention times in the cooking stage are in the order of several hours, typically around 2-5 hours. In
addition, the figures of the application show a residence time in the impregnation stage approximately of the same order as in the cooking stage. However, it is found that while the proposed continuous processes have pulp strength advantages, the cooking stage still has a long retention time and low reaction temperature. This requires huge and expensive digesters designed for, from a technical point of view, high pressures and temperatures.

[0018] In U.S. Pat. No. 3,215,588, and in paper entitled “Rapid alkaline cooking”, Pulp and Paper Magazine of Canada, No 7, pages T-275-T-283, a process is disclosed wherein an extended impregnation stage is utilized followed by a rapid cooking stage. Chip impregnation takes place at a pressure in excess of 10 bar, using cooking liquor. Subsequently, the chips are fed into a continuous digester having a steam zone where the chips are rapidly heated to 170-185° C. and thus cooked before entering a liquid zone where gradual cooling takes place prior to discharge. The paper teaches that the total cycle including impregnation is in the range of 30 to 45 minutes, mostly using impregnation temperatures of 130-150 °C. The process results in pulp relatively low in lignin, having good bleachability according to the standards of the time, and the process is rapid due to the pressurized impregnation stage and the short heating stage. However, the screen rejects content is high and the strength reduced in comparison with pulp produced by conventional methods of the period. These disadvantages are addressed in U.S. Pat. No. 3,644,918, which introduces water saturation of the chips prior to impregnation. By using water-saturated chips, according to U.S. Pat. No. 3,644,918, it is possible to obtain complete and uniform impregnation at atmospheric pressure at e.g. 90° C. within a period of the order of 60 minutes. The whole amount of cooling liquor is added in the impregnation stage. Screen rejects are negligible, the yield is higher and the pulp shows better properties than according to U.S. Pat. No. 3,215,588. However, the authors have found that this process also lacks efficiency, since the water saturation of the chips increases the evaporation demand in the recovery cycle, the reject levels are high and the screened yield is low.

[0019] Thus, development of continuous cooking technology has been characterized by improvements in various fields, e.g. energy efficiency. However, very little attention has been paid to important issues as how to really utilize both “the front-end” and “back-end” of the cooking process to simplify it, retain flexibility and also to improve the pulp quality. The failure to consider these issues has to a great extent been responsible for the development of larger and larger equipment as well as lowering the flexibility of the process and causing lower pulp quality. The development of rapid cooking has failed to understand the conditions which are required to economically produce high-quality pulp.

SUMMARY OF THE INVENTION

[0020] According to the present invention, an improved, continuous alkaline cooking process according to claim 1 is provided, wherein the raw chip material is preheated and air purged, and impregnated with a liquor at a temperature no higher than the boiling point at atmospheric pressure of the impregnation liquor, at retention times of more than 60 minutes. Liquors including fresh cooking liquor are added at an effective alkali concentration in the range from about 0.5 to about 2.2 molal as OH ions; preferably said concentration is in the range from about 0.5 to about 1.5 mol/l as OH/l; more preferably said concentration is in the range from about 0.75 to about 1.5 mol/l as OH/l. A liquid-to-wood ratio in the range of 3 to 10 m³/t odw (m³ per ton oven dry wood) is to be maintained during the impregnation step; preferably said ratio is in the range of 3 to 6 m³/t odw. The impregnated material is subsequently heated to a temperature T2 of at least about 150°C in a time t2 after which follows a cooking stage with a retention time t3, a maximum temperature T3 of no more than 185°C, the liquid-to-wood ratio being at least 2.5 m³/t odw during a substantial part of the heating and cooking step. The total of t2 and t3 shall not exceed 65 min. Fresh cooking liquor is added also during the steps following impregnation. After the cooking step, the delignified material is cooled to a temperature where significant cooking reactions no longer occur.

[0021] In practice, a temperature decrease to about 140°C is sufficient to end the cooking step. Preferably, the time t1 for impregnation is above 120 min, and the temperature T1 in the range from 70°C to the boiling point at atmospheric pressure of the impregnation liquor.

[0022] Preferably, the total of t2 and t3 is in the range from 10 to 50 min, more preferably in the range from 10 to 40 min. Time t2 is preferably not more than 30 min. Further, the liquid-to-wood ratio in a substantial part of the heating and cooking steps is preferably at least 3 m³/t odw.

[0023] Preferably, impregnation takes place at low pressure, for the present purposes defined as up to 5 bar. More preferably, impregnation takes place at about atmospheric pressure. Thus, low pressure equipment may be used, which saves investment costs. Use of pressure may be required to ensure sinking of the chips in the liquid phase. If high pressure equipment is installed it can notably be utilized.

[0024] The average dry-solid of the material entering the impregnation is preferably over 40%; More preferably said dry-solid is over 45%.

[0025] The above process has given excellent results as shown in the examples, and it is a significant process simplification. The differences compared to prior art continuous processes are the unique combination of:

[0026] low temperature during impregnation, which enables low-pressure and temperature equipment in impregnation, and long retention times, i.e. over 60 min

[0027] an alkali concentration of 0.5-2.2 mol OH/l of added liquor in impregnation.

[0028] a short heating and cooking stage (less than 65 minutes) making possible a low volume of cooking equipment in relation to the impregnation equipment.

[0029] a liquid-to-wood ratio over 2.5 m³/t odw and fresh alkali addition in heating and cooking stage or both

[0030] different pressures in the impregnation and cooking stages, which enables simpler equipment for low-pressure impregnation and a lower volume of high-pressure equipment.

[0031] This results in process simplification and great flexibility. Experimental results show that the process gives
high yield, improved bleachability and at least equal quality compared to prior art methods.

[0032] The specific temperatures, effective alkali concentration and times used in a process according to the invention are dependent on the type of wood and the purpose of the product. Hardwoods generally require lower maximum temperatures and effective alkali concentrations compared to softwood. Pulp for unbleached products also normally require lower cooling temperatures than for bleached products. The impregnation time and effective alkali concentration depends mainly on the type of chip. Material hard to impregnate, and consequently requiring longer times, may consist of long and thick chips, or have a large proportion of low-porosity material. The type of equipment and the space available are other factors.

[0033] As raw material for the process according to the invention, industrial wood chips are used. These commonly have an average length above 10 mm, typically 15-35 mm, and an average thickness above 2 mm, typically 3-7 mm.

[0034] According to one aspect of the invention, a plant for carrying out the method comprises a digesting system including an impregnation vessel and a digester in fluid communication with the impregnation vessel. The volume V of the impregnation vessel is larger than 1/1, preferably larger than 2/1, more preferably larger than 3/1 of the volume of the digester.

[0035] The system may comprise a first transfer line between the impregnation vessel and the digester for transporting the impregnated material to the digester, a separator, comprising a withdrawal space, disposed in connection with the first transfer line for separating a transport liquid from the impregnated material; a first return line connected to the separator to conduct the transport liquid from the separator back to the first transfer line; a second return line attached to the first return line and to the impregnation vessel for supplying a portion of the transport liquid to an inlet of the impregnation vessel; a supply line connected to a supply space adjacent the level of the material in the impregnation vessel.

**DISCLOSURE OF THE INVENTION**

**BRIEF DESCRIPTION OF THE DRAWINGS**

[0036] FIG. 1 is a graphic representation of the time-temperature profile of prior art conventional batch cooking,

[0037] FIG. 2 is a graphic representation of the time-temperature profile of prior art displacement batch cooking,

[0038] FIG. 3 is a graphic representation of the time-temperature profile of prior art continuous cooking of the Kamyr type,

[0039] FIG. 4 is a graphic representation of the time-temperature profile of an embodiment of the invention,

[0040] FIG. 5 is a flowchart showing an embodiment of the invention,

[0041] FIG. 6 is a schematic representation of the water balance of a traditional cooking system,

[0042] FIG. 7 is a schematic representation of the water balance of a cooking system in accordance with the present invention,

FIG. 8 shows the brightness achieved versus consumption of active chlorine in pulps prepared using the conditions set forth in Table 1.

FIG. 9 shows the brightness as a function of viscosity of pulps prepared according to the examples in Table 1.

FIG. 10 shows the active chlorine consumption against bleached yields in the examples according to Table 1, and

FIG. 11 shows the reject percentage as a function of impregnation time in pulp cooked to two kapa numbers according to the invention.

**DETAILED DESCRIPTION**

[0047] FIGS. 1 to 4 show the temperature profiles of prior art pulping methods as well as that of the present invention. Referring now to FIG. 1, which shows the temperature curve against time in a conventional batch cook, region 1 of the curve represents the heat-up phase, region 2 illustrates cooking at about the maximum temperature and region 3 illustrates the discharge and cooling of the conventional batch digester.

[0048] Typically, the duration of region 1 is 60 to 150 minutes, and that of region 2 60 to 120 minutes. The sum of region 1 and 2 is typically about 150 minutes.

[0049] In FIG. 2 showing the corresponding curve of a prior art displacement batch process, region 5 represents the impregnation phase, region 6 the hot liquor fill phase, i.e. hot black liquor treatment and hot white liquor charge, region 7 represents the temperature adjustment phase, usually carried out by circulating the digester content and heating, region 8 illustrates the cooking phase at cooking temperature. Region 9 represents the displacement with cool wash liquid and region 10 represents the cold discharge.

[0050] Typically, the duration of region 5 is typically about 30 minutes, but it can be 10 to 40 minutes depending on digester size, at a temperature below 100° C. Region 6 is typically about 30 minutes (can be 15 to 40 minutes depending on digester size). Regions 7 and 8 are typically 65 to 100 minutes. Thus, regions 6, 7 and 8 together typically represent 95 to 130 minutes. Region 9 is typically 45 minutes (can be 20 minutes to 60 minutes depending on, among other factors, digester size).

[0051] In FIG. 3, illustrative of a prior art continuous process, region 11 represents the impregnation phase, region 12 represents heating and region 13 represents a cooking phase, which can occur in both concurrent and countercurrent modes. Region 14 represents displacement and cooling of the cooked material before discharge from the digester. Region 11 is typically 30 to 60 minutes or shorter at a temperature of 115-130° C. Regions 12 and 13 are over 90 minutes, typically 240 to 360 minutes.

[0052] FIG. 4 shows the advantageous temperature profile of the present invention. Region 20 represents the impregnation phase, which as can be seen is substantially extended relative to processes presently in use. Region 21 represents the heating-up phase. Region 22 represents the short reaction time and region 23 the cooling of the cooked material. Curve 24 illustrates schematically an optional cooling and
washing method. Between regions 20 and 21, feeding of the pre-impregnated material to the cooking reactor takes place.

**[0053]** FIG. 5 is a flowchart of a process according to the invention. Lignocellulosic material 30 in the form of industrial wood chips enters a pre-steaming phase 31. Steam 36 is supplied to heat the chip flow and purge air therefrom, and gases 37 are removed from phase 31, preferably to a closed system. In pre-steaming, the chips are heated to a temperature of 80-120°C, preferably 95-110°C. The retention time at the above temperature should preferably be 5-40 min. Pre-steamed material 38 enters the impregnation stage 32. The transfer method and equipment in point 38 between phase 31 and 32 depends on the counter-pressure in stage 32. The residence time of the stage 32 is at least 60 minutes. It can be significantly longer, depending on the available size of equipment. Longer times or impregnation times of more than about 24 h may be used for example when combining the impregnation stage with chip storage between the chipping unit and the cooking plant. The impregnation time rarely exceeds 120 hours in the same equipment. The impregnation equipment may be a down-flow vertical vessel or a horizontal conveyor type vessel with at least one inflow and at least one outflow point for the material, known to the person skilled in the art. Installed continuous digester vessels can be used e.g. when upgrading an existing plant. When using longer retention times, the impregnation device can be considered to be of the chip silo vessel type. Several vessels can be used in series or in parallel. According to the invention, the impregnation vessels are preferably dimensioned for a low pressure, i.e. pressure in the area from about 0 to 5 bar, more preferably, the vessels are dimensioned for pressure in the range from atmospheric to about 1 bar. High-pressure equipment (over 5 bar design pressure) can also be used when for example upgrading a plant to a process according to the invention. Fresh alkali 39 is added to the stage. The amount may be, for example, 30 percent or more of the total fresh alkali to be added calculated as total titratable alkali (TFA) per charged unit of wood, but additional fresh alkali is invariably added in the cooking stage. Spent liquor 46 is added as needed, recycled from e.g. a subsequent liquor-separation stage. The effective alkali concentration of the added liquors is 0.5-2.2 mol OH/l; preferably 0.5-1.5 mol OH/l.

**[0054]** The fresh alkali 39 and spent liquor 46 can be added together at one addition point, or in sequences during the impregnation. Spent liquor can be added first, and then fresh alkali is added and some spent liquor withdrawn. Fresh alkali can also be added first and then spent liquor. Parts of spent liquor and fresh alkali can also be added first, and then fresh alkali is added together with some withdrawal of spent liquor. The fresh alkali used can be both causticized liquor, normally referred to as while liquor, and uncausticized liquor, normally referred to as green liquor, or also derivatives of the above mentioned liquors, e.g. a mother liquor from crystallization of sodium carbonate from green liquor.

**[0055]** Impregnated material 40 leaves the impregnation reactor via a transfer system 41, which may be one of various combinations of discharge systems in the outlet part of the impregnation vessel and feeding technology known to the person skilled in the art. The system is supplied with liquor 45 as required e.g. for dilution. Transfer systems to be used are pumps, chamber feeders (e.g. of the high-pressure (HP) feeder type), screws, scrapers and injectors etc., and combinations thereof, known to the person skilled in the art. The stream may enter a device 43 for liquid removal, which device may be a screen or a liquor separator of a known type. In device 43, the free liquor 48 is removed and the liquor is separated from the chips. The chips are adjusted to a predefined value, after which the chips 44 enter the cooking stage 33. The remaining required amount of fresh cooking liquor 47 is added to the cooking stage 33 together with spent liquor 60 in order to e.g. adjust the alkalinities in liquor-to-wood ratio in cooking stage 33. The liquor-to-wood ratio is at least 2.5 m³/ton dw. In practice, the liquor-to-wood is always below 100 m³/ton dw. The temperature of the liquor may require adjustment to hold the preferable temperature between 70°C and atmospheric boiling point.

**[0056]** The digester used must be able of heating the chips to the cooking threshold temperature T2, preferably within about 30 minutes. This can be accomplished by direct steaming and/or by pre-heating the added liquors. In practice the heating time t2 is always above about 1 minute.

**[0057]** After the threshold temperature T2 is reached, exothermic reactions and possible additional heating raise the temperature to a maximum temperature T3. The total time of heating and cooking at maximum temperature must not exceed 65 min. The cooking zone can be a vertical or horizontal reactor with at least one input and one output of material. A vertical reactor can be fed from either top or bottom, i.e. down-flow or up-flow reactor or a combination of down-flow and up-flow reactor. A horizontal reactor can be equipped with a conveyor device, as a chain conveyor or a screw. The cooking zone can also be a reactor inclined at a certain angle, e.g. 45°. The cooking reactor can be equipped with liquor and steam addition points and with withdrawal points for both gas and liquors. These addition and withdrawal points can be several. At the end of cooking, the effective alkali concentration of the cooking liquor can be 0.05-0.7 mol OH/l, preferably in the range 0.1-0.5 mol OH/l. The cooked material 53 exits the cooking stage and enters a washing and cooking phase 34, in which cooler liquor is introduced to essentially end the cooking reactions, leach out dissolved material inside the cooked chips, and wash the cooked material. Any suitable combination of displacement, liquor withdrawal, dewatering and dilution may be used. Technically, this may be accomplished using a liquor displacement and withdrawal zone in the cooking reactor, dilution of the cooked material, dewatering and displacement stages occurring outside the cooking reactor in a subsequent vessel, or pulp washing equipment known to persons skilled in the art. The washing stage is supplied with a substantially aqueous liquid, which can be e.g. water, condensate or suitable diluted process liquor. Spent liquor 52 from the washing stage may be re-circulated 60 to the digester, preferably through a heating device (not shown) and possibly mixed with an additional liquor which may be a liquor from the impregnation stage, e.g. from point 55. Spent liquor 56 and 57 from the cooking and washing stages, respectively, is removed from the process. The heat of the combined liquor 62 is recovered either separately of at least one of the liquors representing liquor 62, i.e. liquors 56, 57 and 58, or of the combined liquor 62. The recovery of heat can take place by flashing or heat exchange in one or several stages. The recovered heat can be used to heat the material 30 in the pre-steaming stage 31, and/or the recovered heat can
be used to heat the fresh alkali 47 or the spent liquors 60, 52 or 59, to heat other process liquids, e.g. in evaporation, or to heat water.

[0058] The liquor balance of the process may further include circulation of liquor 48 and 49 from the separation device and from any phase of the impregnation stage, respectively, to the beginning of the impregnation stage. Liquor 55 from the impregnation stage may also be conducted to the chemicals recovery through line 58.

[0059] The washed material 61 proceeds to further treatment stages as e.g. delignification, bleaching, refining, screening etc. depending on the purpose of the final product.

[0060] FIG. 6 shows a typical water balance of a prior art process, compared with the water balance of a process according to the present invention in FIG. 7. It can be seen, that the method according to the invention does not affect the water balance of the plant, but the difference in relation to conventional cooking is clear. A substantial part of the white liquor is added to a pre-treatment stage to completely impregnate the chips prior to cooking. In addition, some residual liquor after the pre-treatment is re-circulated to the liquors introduced in the impregnation to dilute the EA of added liquors and thereby avoid too strong liquors being applied on the wood chips, which may lower the quality of the pulp. Thus, because of the efficient pre-impregnation, a minor amount of white liquor, that is, small compared to conventional cooking, is added to the cooking stage to adjust the ability of the cooking stage. In conventional cooking, substantially all white liquor is added to the cooking stage. Therefore, conventional cooking with minor pre-impregnation lacks efficiency in pre-impregnating the chips prior to heating and cooking the chips.

[0061] Table 1 shows the results of, on the one hand, comparative laboratory cooking experiments (1-4) using various typical conditions for prior art continuous and batch cooking, and on the other hand experiments (5-11) using conditions according to the present invention.
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<th>Example</th>
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<td>30</td>
<td>4320</td>
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<td>Heating and Cooking</td>
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<tr>
<td>EA of added liquor, mol OH/ℓ</td>
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<td>160</td>
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<td>End-of-cook residual EA, mol OH/ℓ</td>
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<td>7</td>
<td>8</td>
<td>9</td>
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<td>1014</td>
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<td>979</td>
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<td>D(EOP)DnD</td>
<td>D(EOP)DnD</td>
<td>D(EOP)DnD</td>
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<td>90.5</td>
<td>92.0</td>
<td>91.7</td>
<td>91.5</td>
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<td>92</td>
<td>92.7</td>
<td>nd</td>
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<td>nd</td>
<td>9.0</td>
<td>11.1</td>
<td>10.6</td>
<td>10.7</td>
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<td>nd</td>
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</table>
[0062] The following abbreviations are used in the examples:

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<tr>
<th>Abbreviation</th>
<th>Definition</th>
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<tbody>
<tr>
<td>EA</td>
<td>Effective alkali = NaOH + 1/3Na2S, expressed as NaOH equivalents</td>
</tr>
<tr>
<td>nd</td>
<td>not determined</td>
</tr>
<tr>
<td>ECF</td>
<td>Elemental Chlorine Freee</td>
</tr>
<tr>
<td>D</td>
<td>Choline dioxide bleaching step</td>
</tr>
<tr>
<td>n</td>
<td>neutralization</td>
</tr>
<tr>
<td>(DnD)</td>
<td>Choline dioxide bleaching step with intermediate neutralization</td>
</tr>
</tbody>
</table>

**EXAMPLES 1 and 2**

[0063] Mill-scale production according to prior-art Kamyr continuous cooking of industrial eucalyptus chips to kappa numbers typical of eucalyptus cooking. Sampled pulps were oxygen delignified and ECF bleached in the laboratory. Bleaching chemicals demand for a given pulp brightness was determined and the pulp strength measured by beating and testing.

**EXAMPLE 3**

[0064] Production of eucalyptus pulp according to prior-art process disclosed in U.S. Pat. No. 3,664,918 (vapor phase pulping of water saturated lignocellulosic materials) and example 1 of U.S. Pat. No. 3,664,918.

[0065] The industrial eucalyptus chips (5.5 kg oven dry weight) were first submerged in water overnight at 2 bar overpressure and room temperature. The excess water was separated. The water saturation resulted in chips of 44.6% dry solids. The water-submerged chips were metered into a chip baskled positioned in a 32-liter jacketed displacement digester with liquor circulation. The chips were impregnated with white liquor (EA charge of 33.7% NaOH calculated on wood, EA 122 g NaOH/1 and sulfidity 30%) at a liquor-to-wood ratio of 4 m³ per ton of dry wood at 90°C, 60 minutes and atmospheric pressure. After impregnation of the chips and removal of excess liquor, the impregnated chips were then subjected to steaming and the temperature of the chips was initially raised to 100°C for 20 min and subsequently treated at 175°C for a total of 36 min, including heating-time of 7 minutes. After cooking the digester content was cooled with water. After the cook the pulp was wet disintegrated and screened. Kappa number, yield, reject, brightness were determined on the cooked pulp.

**EXAMPLE 4**

[0066] Production of normal eucalyptus pulp.

[0067] Example 4 show laboratory simulation data of a process simulated according to prior-art displacement batch cooking of industrial Eucalyptus.

[0068] 4.5 kg eucalyptus chips (oven dry basis) were metered into a chip basket positioned in a 26-liter jacketed displacement digester with liquor circulation. The same chip raw material as shown in Example 3 was used. The chips were pre-steamed for 10 minutes at 100°C. Then impregnation liquor fill was conducted with an impregnation liquor containing 0.29 mol OH/1 of EA. After 30 minutes impregnation, hot black liquor treatment occurred for 20 minutes with a HHL containing 0.205 mol OH/1 of EA and a temperature of 148°C. Then hot white liquor (105 g NaOH/1 as EA, sulfidity 40%) at a charge of 11.6% as NaOH (EA) was added for 10 minutes. The digester content was then heated for 20 minutes to the cooking temperature of 160°C. The time at cooking temperature was 41 minutes. After the cook the pulp was wet disintegrated and screened. Kappa number, yield, reject, brightness were determined on the cooked pulp. The cooked pulp was then oxygen delignified and ECF bleached in the laboratory. Bleaching chemicals demand for a given pulp brightness was determined and the pulp strength measured by beating and testing.

**EXAMPLE 5**

[0069] Production of eucalyptus kraft pulp in accordance with an embodiment of the present invention.

[0070] 5.5 kg of oven dry eucalyptus chips was metered into a chip basket in a 32-liter jacketed displacement digester with liquor circulation. The same chip raw material was used as in Examples 3 and 4. The chips were first pre-steamed at 100°C for 30 minutes. Impregnation occurred for 3 days at a temperature of 95°C and a small overpressure of 0.5 bar. The alkalinity of the added liquor was 1.25 mol OH/1 and the liquor-to-wood ratio was 4.6 dm³ per kg of dry wood. The added impregnation liquor contained white liquor at a sulfidity of 40% and spent liquor drained from previous impregnations using the same process. After impregnation of the chips and removal of excess liquor, pre-heated cooking liquor at various alkali concentrations (EA) was added for 5 minutes and the liquor-to-wood ratio was simultaneously adjusted to 3.5 m³ per ton of dry wood. The digester content was heated to the cooking temperature in about 10 minutes and the temperature was kept at temperature for 10 minutes. After cooking, the digester content was cooled and the liquor was drained. After the cook the pulp was wet disintegrated and screened. Kappa number, yield, reject, brightness were determined on the cooked pulp. The cooked pulp was then oxygen delignified and ECF bleached in the laboratory. Bleaching chemicals demand for a given pulp brightness was determined and the pulp strength measured by beating and testing.

**EXAMPLE 6**

[0071] The experiment was carried out as disclosed in Example 5, but the impregnation time was 60 min and the cooking conditions were adjusted to give about the same kappa number as in Example 5.

**EXAMPLE 7**

[0072] The experiment was carried out as disclosed in Example 6, but the cooking conditions were adjusted to give a higher cooking kappa number.

**EXAMPLE 8**

[0073] The experiment was carried out as disclosed in Example 7, but the impregnation time was adjusted to 180 minutes and the cooking conditions were adjusted to give slightly higher kappa number than in Example 7.

**EXAMPLE 9**

[0074] The experiment was carried out as disclosed in Example 8, but the impregnation time was adjusted to 3 days and the cooking conditions were adjusted to give slightly higher kappa number compared to Example 8.

**EXAMPLE 10**

[0075] The experiment was carried out as disclosed in Example 6, but the impregnation pressure was adjusted to 10
bar and the cooking conditions were adjusted to give slightly higher kappa number compared to Example 6.

EXAMPLE 11

[0076] The experiment was carried out as disclosed in Example 8, but the impregnation pressure was adjusted to 10 bar and the cooking conditions were adjusted to give slightly lower kappa number compared to Example 8.

[0077] Table 1 shows the cooking characteristics, unbleached pulp results and the subsequent oxygen delignification, ECF bleaching and PFI beating results. All oxygen delignifications, ECF bleachings, PFI beatings and tests were performed in the laboratory.

[0078] Based on the results in Table 1 and FIGS. 8 to 11, the present invention offers the following surprising benefits over a state-of-the-art cooking process:

[0079] By using over 60 minutes pre-impregnation below 100°C, using 0.5-2.2 mol OH/l of added liquors and when cooking at a liquor-to-wood ratio over 2.5 as shown in examples 5 through 11:

[0080] remarkably shorter residence time in heating and cooking can be used compared to over 1.5 hours in prior-art continuous digesters

[0081] the required cooking volume is considerably reduced

[0082] the unbleached and oxygen bleached pulp is brighter pulp at same kappa number

[0083] lower or equal rejects amounts at same or higher kappa number. In a process according to the invention and according to the methods described, the reject level depends on the impregnation time and kappa number target (see FIG. 10 showing reject levels of pulps at kappa numbers 20 and 25 and impregnation times of 0-3 days using a retention time of 25 minutes in heating and cooking).

[0084] the reject level is independent on impregnation pressure in the range 0.5 bar to 10 bar for pre-steamed chips implementing that low-pressure impregnation equipment can be used in impregnation

[0085] higher unbleached screened yield

[0086] higher kappa number reduction in oxygen delignification. Example 7 used more NaOH in oxygen delignification, but the additional cost for this is minor since oxidized white liquor from the recovery cycle, i.e. low-cost NaOH, is usually used in oxygen delignification.

[0087] considerable lower active chlorine chemical consumption in ECF bleaching by about 50-65%

[0088] bleached pulps gives a pulp of higher viscosity, see example 5,

[0089] higher bleached yield at lower bleaching costs.

[0090] higher tensile strength although the brightness of the pulps are higher

[0091] The effect of impregnation time is demonstrated in FIG. 11. The reject percentage is shown as a function of impregnation time as pulp is cooked according to the invention to kappa numbers 20 and 25 using a total heat-up and cooking time of 25 min. It is seen, that a satisfactory level is reached when 1 hour impregnation residence time is used; a further half percent decrease is achieved by extending impregnation with a further hour. The improvement due to extension even to three days is marginal. However, table 1 shows that the bleaching chemical consumption is significantly lower and bleached pulp viscosity is higher when using 3 days impregnation compared to 60 minutes impregnation.

1. A continuous, alkaline process for the production of pulp from wood chips, said process comprising the steps of:

impregnating the chips;

heating the impregnated chips to a temperature of at least about 150°C. and cooking the chips, the maximum temperature of the cooking step being less than 185°C. and the total time of the heating and cooking steps being less than 65 min, during which period fresh alkali is added as well as possible additional liquor, so as to result in a liquor to wood ratio of at least 2.5 m³/t owd during a substantial part of the heating and cooking steps, and

cooling the cooled material to a temperature where cooking reactions essentially cease.

2. A process according to claim 1, wherein at the most 90% of the total fresh alkali calculated as total titrable alkali per unit charged wood is added during the impregnation step.

3. A process according to claim 1 or 2, wherein the time t₁ of the impregnation step is above 120 min.

4. A process according to any claim 1-3, wherein the total of time t₁ of the heating step and the time t₃ of the cooking step is in the range from 10 to 50 min, preferably in the range from 10 to 40 min.

5. A process according to any claim 14, wherein the time t₂ of the heating step is below 30.

6. A process according to any claim 1, wherein the liquid-to-wood ratio is in the range from 3 to 6 m³/t owd during a substantial part of the impregnation step.

7. A process according to claim 1, wherein the liquid-to-wood ratio is at least 3 m³/t owd during a substantial part of the heating and cooking steps.

8. A two-vessel digesting system for performing the method according to claim 1, comprising an impregnation vessel and a digester in fluid communication with the impregnation vessel, whereby the volume V of the impregnation vessel is larger than 1/1, preferably larger than 2/1, more preferably larger than 3/1 of the volume of the digester.

* * * * *