(57) Abstract: The invention concerns a method during the cooking of cellulose pulp in which the raw material, preferably in the form of chips, undergoes a successive elevation of temperature towards the cooking temperature while the chips are first impregnated with an impregnation liquor followed by cooking in a cooking liquor that is preferably alkaline. Through the addition of pre-evaporated black liquor, either as early as the impregnation stage or during the subsequent cooking stage, an improved delignification process is achieved in which the pulp obtains improved properties, primarily with respect to strength/viscosity for a given degree of delignification, and also with respect to yield. The method can be used both for continuous cooking and for batchwise cooking of cellulose pulp.
For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.
Cooking of cellulose pulp in a cooking liquor containing pre-evaporated black liquor

The present invention concerns a method for the production of cellulose pulp according to the introduction of claim 1.

The Prior Art
The technology of cooking has undergone significant development for several decades. During the 1960s and 1970s, systems were used in which essentially all liquor for cooking, including white liquor, were added in batches at the pre-treatment stage. Cooking in continuous digesters subsequently took place in the same liquor down through the digester, and the liquor was then withdrawn. Relatively high levels of alkali were established at the start of the cooking stage, such that sufficient alkalinity was be maintained throughout the complete cooking stage. It became clear that the high levels of alkali at the beginning of the cooking stage were detrimental to the quality of the pulp, and this led to several variations on this system being suggested. These variations include such techniques as MCC (modified continuous cooking), ITC (isothermal cooking) using the same cooking temperature throughout the digester, and EMCC (extended modified cooking).

Later, during the 1980s, black liquor impregnation, in which liquor used in the cooking stage is reintroduced to constitute part of the impregnation liquor, was developed for both batchwise cooking and for continuous cooking. In this case, white liquor could be added batchwise at the end of the impregnation, or at the beginning of the cooking stage. The used cooking liquor, i.e. the black liquor, that was withdrawn from the cooking stage could in this case have a relatively high residual alkali content, on which this withdrawn black liquor was reintroduced to the impregnation. Here, most of the residual alkali was consumed before the used impregnation liquor was sent to recovery following withdrawal (concentration by evaporation and soda recovery furnace). This technique allowed a lower level of alkali to be established at the beginning of the cooking stage.
Several solutions are known in which black liquor is used as an impregnation liquor in an impregnation zone before cooking. A system is revealed in US 5,080,755 that has black liquor in the input. A variant is revealed in US 5,053,108 in which black liquor withdrawn from the digester is recycled to the high-pressure layer in order to there form the major part of the treatment liquor in the transfer circulation for the digester. A variant that has been developed further is revealed in EP477059, in which wood chips impregnated with black liquor are raised to cooking temperature before the main addition of white liquor. These show that many different suggestions for process have been studied, with the aim of improving the quality of the pulp while at the same time maintaining the high degree of delignification in the pulp that is washed after cooking.

Marketing by Andritz-Ahlstrom of another cooking technology, denoted by LO-SOLIDS, began during the 1990s. This involves the continuous withdrawal of cooking liquor with a high degree of organic material and the replacement of this by new or treated cooking liquor that has a lower level of dissolved organic material (DOM/dissolved organic material). However, this system suffers from the disadvantage that the cooking process often results in a lower yield, caused by the fact that with the withdrawn organic material, such as lignin, also contains carbohydrates, principally hemicellulose.

Other methods of improving the cooking stage, both with respect to yield and with respect to pulp quality, are the addition of polysulphide, AQ or dissolved Xylan.

The yield is highly significant during cooking since an increase of only 1% means that a production facility of normal size, having an output of 1,500 tonnes a day, would experience an increase in production of 15 tonnes, which, with a pulp price of 700 USD/ADT, gives an increased income of 10,500 USD a day.
The Aim and Purpose of the Invention

Subject to the developments in cooking technology, principally the technology used in continuous cooking, two dominating technologies are currently available. These are the technique known as "LO-SOLIDS" and the technique developed by Kvaerner Pulping AB known as "COMPACT COOKING". Extremely high liquor/wood ratios are established during COMPACT COOKING in the initial phases of the cooking stage, with a very high level of black liquor present in the cooking liquor.

It has now become clear, surprisingly, that a very favourable delignification is established during the cooking stage if black liquor is present in the cooking liquor. This contrasts strongly with the principles of the LO-SOLIDS technology. Thus, cooking liquor with a high level of dissolved organic material is not withdrawn, and replaced with cooking liquor with a lower level of dissolved organic material, as it is in the LO-SOLIDS technology.

The problem is rather the reverse: it is desired to increase the fraction of dissolved organic material while at the same time maintaining the levels of other added liquors, white liquor, etc., with respect to amount and concentration. It is particularly desired to enrich the cooking liquor with the organic material that advantageously influences delignification and that also contributes, to a certain extent, to an increased yield.

The relevant type of desired supplement for the cooking liquor is already available at the pulp mills, but it is present in the evaporation stage before the recovery. Nobody has yet realised that a partially evaporated black liquor has an advantageous effect on the cooking stage, nor have they realised that the partially evaporated black liquor should be returned to the cooking stage from the evaporation stage.

The main aim of the invention is to increase the selectivity in the cooking stage by accelerating delignification. This results either in the achievement of improved pulp quality (viscosity/pulp strength) and higher yield at the same
degree of delignification (reduction in kappa value), or in the achievement of a higher degree of delignification at the same pulp strength and yield.

The invention also allows a positive increase in OH⁻ and in HS⁻ ions during the cooking stage, something that in a known manner gives better selectivity and bleachability for the cellulose pulp produced.

Another purpose is the exchange of the improved delignification effect during the cooking stage for at least one of the following advantages:

- Smaller continuous digesters, that is: cheaper systems for a given production volume (shorter stoppage times);
- Increased production capacity for both batchwise and for continuous cooking, with a maintained pulp quality;
- Reduced need for cooking chemicals (alkalis), something that gives lower production costs.

The invention can be used on both steam-phase digesters and on hydraulic digesters; with inverted top separators, with downward-feeding top separators and with types that lack a top separator; and it can be used during the production of cellulose pulp using both the sulphite process and the sulphate process. In the same way, deciduous wood, coniferous wood, annuals (such as bagasse, etc.) and others can constitute the source of cellulose.

The invention can be used with batchwise cooking, in which the chips are fed into a vessel in which a sequential treatment with various impregnation liquors and cooking liquors subsequently takes place on the chips that are held stationary in the vessel.

The invention can also be used in continuous digesters, in which pre-evaporated black liquor is added to cooking liquors that pass either upstream or downstream with the chips during the cooking stage, at the beginning of the cooking stage, in the middle, or at its end.
Description of Drawing
Figure 1 shows the principles of application of the invention in a cooking system.

Detailed Description of Preferred Embodiments
Figure 1 shows schematically a cooking system with the associated recovery of cooking chemicals. The chips are first handled in a chip management system 1 (Chip) in which the chips can preferably be made basic with steam for the expulsion of air and for the first warming of the chips. A first addition of the process liquid occurs here such that the chips become mixed into a chip/liquid mixture.

Black liquor BL1, which has been withdrawn from a subsequent cooking stage, is added at a subsequent impregnation stage (BL-Imp). The black liquor can be added with the chips at the start of the impregnation stage and it can accompany the chips in what is known as downstream treatment, after which the black liquor is withdrawn from the impregnation vessel. This black liquor BL2 normally has a relatively low level of alkali of about 5-20 g/l, and it can, after a pressure reduction in a cyclone/pressure reduction vessel FL, be sent for recovery. The pressure is normally reduced down to a pressure level that does not exceed an excess pressure of 0.5 bar, and often to a pressure that is essentially atmospheric pressure. The cooking process takes place at an elevated pressure, normally with an excess pressure of 5-25 bar in the digester, and a preceding impregnation with black liquor can either take place under pressure or at atmospheric pressure.

The treated chips after impregnation with black liquor are transferred to the digester (Cook) where cooking liquor Cliq is added. The chips are cooked during the cooking stage at a cooking temperature that lies in the interval 150±20°C, after which the cellulose pulp is transferred for further delignification and bleaching (Bleach), preferably passing through an intermediate washing stage (not shown in Figure 1).
Recovery consists, in a conventional manner, of a number of evaporation stages 5a-5e in which the black liquor, which is at essentially zero excess pressure, (BL2 after passing through FL) is subject to an evaporation in several stages from an initial level of dry matter content TS in the black liquor (BL2) of 17-20%, to a level that lies over 70-80%. The evaporation line 5a-5e consists of a number of evaporation stages, know as effects, that the black liquor passes through in sequence, while heating steam is passed in the opposite direction to the flow of black liquor. Normally, the earliest and hottest steam is used in the evaporation stage that treats the black liquor with the greatest level of dry material, that is, the final stage seen from the point of view of the flow of black liquor. Expelled volatile substances are also obtained at each evaporation stage, and these are dealt with by special gas management systems (not shown in Figure 1) or by turpentine recovery systems, or they are led to other stages in order there to be mixed with the heating agent (the steam).

Superconcentrators 6 may be included as a last stage of the evaporation, before the black liquor is combusted in a soda recovery furnace 7. A melt is formed in this furnace that is removed from the bottom (as shown in Figure 1), and that is suspended to form green liquor (not shown) and sent to a causticization plant where white liquor is reformed.

Naturally, in contrast to what is shown in Figure 1, the first evaporation stage can be constituted by a specially designed evaporation stage that only evaporates that amount of black liquor that is to be recycled to the impregnation stage or the cooking stage. Such a stage can thus be located next to the digester and does not necessarily need to be arranged in association with the other evaporation stages prior to the soda recovery furnace.

According to the invention, a portion of the partially evaporated black liquor PV_BL is thus removed from, for example, the first stage 5a and led back to the cooking stage. As is shown schematically in the figure, the pre-evaporated black liquor PV_BL can be added to a digester circulation in which cooking liquor is withdrawn from the cooking stage, normally through strainers in the
wall of a continuous digester, and is then returned to the centre of the digester through a central pipe placed at the same height as the strainers. In this way, the pre-evaporated black liquor will become mixed with other treatment liquor before it is added to the cellulose material before the latter is cooked at the actual delignification stage. The pre-evaporated black liquor can, in such an addition process, be added at a location in the digester at which the bulk delignification stage starts.

The pre-evaporated black liquor can, in one alternative, be added to the cooking liquors CLiq that are to be added to the digester before the cooking stage. This alternative is shown using dashed lines in Figure 1. In this way, the pre-evaporated black liquor can be mixed with other cooking liquors before addition to the digester, and at such an early stage that the pre-evaporated black liquor constitutes part of the cooking liquor at the initial delignification stage of the cooking stage.

The invention can be modified in a number of ways within the framework of the claims.

For example, the invention can also be used during batchwise cooking of chips that have been filled into the vessel, following the sequence:

1) Filling the vessel with chips
2) Heating the chips with steam
3) Heating/impregnation with warm black liquor
4) Heating/impregnation with hot black liquor
5) Cooking with cooking liquor
6) Cleaning compression following the cooking stage, in which the expelled cooking liquor is stored in tanks for hot black liquor
7) Cleaning compression following the previous stages with cleaning liquor, where the liquid expelled first is stored in tanks for warm black liquor.

Emptying of the cooked and washed chips.

In this type of cooking sequence, the pre-evaporated black liquor can be added in batches to the cooking liquor that is added to the chips in step 5 above. Alternatively, a modified impregnation stage according to step 4 above can be used, in which the pre-evaporated black liquor is added in batches to the hot black liquor added in step 4, alternatively the purging of the vessel with
pre-evaporated black liquor as a conclusion of step 4, in order to expel residual amounts of hot black liquor that have not been enriched with pre-evaporated black liquor. As a further alternative, the pre-evaporated black liquor can be added in batches to the cooking stage during the commencement of step 5, whereby the pre-evaporated black liquor is included into a digester circulation for mixture with the cooking liquor that remains in circulation in the vessel during the cooking stage.

The pre-evaporated black liquor consists, according to the invention, of a heat-treated black liquor that has a content of dry matter (TS) that exceeds the level of dry matter that can be obtained in the black liquor that is withdrawn from the process and whose pressure is subsequently reduced. This is equivalent to the black liquor that is denoted by BL2 in Figure 1 and whose pressure is reduced in at least one pressure reduction vessel, FL in Figure 1. The content of dry matter TS in this liquor can normally lie around 17-20%, and the content of dry matter TS of the pre-evaporated black liquor is, according to the invention, to be raised by at least 10% from this level, to 27-30%.

An improved effect of the delignification process is obtained even at this modest increase in the content of dry matter. The black liquor is preferably evaporated further to a content of dry matter of at least 30-40%, and preferably at least 50%. The higher the content of dry matter, the less the optimal liquor/wood ratio will be affected during the cooking stage without having to reduce the necessary batchwise addition of other cooking liquors.

The black liquor that, according to the invention, is pre-evaporated can be constituted by pressurised black liquor that has been directly withdrawn from the digester or indirectly withdrawn through a black liquor impregnation, which may be either under pressure or essentially at atmospheric pressure, and subsequently pre-evaporated. The black liquor may also be constituted by such black liquor that has passed through a reboiler, in which the black liquor is first used to generate steam before being pre-evaporated in the manner according to the invention.
Twice as much recirculated pre-evaporated black liquor with a content of dry matter of 27-30% is normally required than would be required if the content of dry matter was around 60%, given similar conditions in the digester with respect to other parameters.

The amounts that are available for return to the cooking stage depend on:
- the current content of dry matter in the pre-evaporated black liquor
- the current liquid/wood ratio during the cooking stage
- the amount of black liquor that accompanies the chips from a preceding black liquor impregnation
- the current raw material (deciduous wood, coniferous wood, annuals, eucalyptus, etc)
- the required batchwise addition of white liquor and its concentration of alkali
- the other types of cooking chemicals that are added.

When liquid/wood ratios that lie at the lower end of the range, around 3-3.5:1, i.e. 3 to 3.5 m$^3$ liquid for each cubic metre of chips, the amount of pre-evaporated black liquor with a content of dry matter around 40% should exceed at least 5% of the total liquid amount, which corresponds to an amount of pre-evaporated black liquor of around 0.15-0.175 m$^3$ for each cubic metre of chips, in order for an appreciable effect of the delignification to be achieved. At higher liquid/wood ratios during the cooking stage, from around 7:1 and up towards 8:1, an equivalent increase of the minimum amount of pre-evaporated black liquor that may, at the most, be required is around 0.35-0.40 m$^3$ per cubic metre of chips, in order to achieve an appreciable effect on the delignification. If the increased liquid/wood ratio in the cooking zone is established by internal recirculation of the cooking liquor, the required increase for an effect on the delignification will be smaller.

Thus, relatively modest amounts of pre-evaporated recirculated black liquor are sufficient that in normal cases amount to a few percent of the total amount of liquor in the cooking stage.
As much as 20-40% of the total amount of liquor can be constituted by pre-evaporated black liquor in cooking situations in which the cooking process has been established with a very low fraction of residual black liquor from the impregnation.

The invention, however, is not uniquely determined by the amounts that are recycled since, as has been previously mentioned, other process parameters during the cooking stage can influence the amount required, as can the type of cellulose pulp (deciduous wood, coniferous wood, annuals, etc.) that is being cooked.

The invention is based on the principle of returning, in contrast to other cooking methods, dissolved organic material to the cooking process, which released organic material has been enriched by initially undergoing substantial evaporation with the purpose of increasing the content of dry matter in the black liquor. Thus, the concentration of the organic material increases, which has surprisingly turned out to influence the delignification process in an advantageous manner and to contribute to increased yield, principally due to the hemicellulose in the pre-evaporated black liquor being reprecipitated onto the cellulose fibres.

It is important for obtaining the best effect that the pre-evaporated black liquor is present during the main part of the bulk delignification stage, more than 50% of the retention time of the chips in the bulk delignification stage, and preferably during the initial delignification stage.

The recycled pre-evaporated liquor can be further heat-treated in a separate stage and/or certain fractions can be mechanically separated, and it can be adjusted with respect to the levels of other chemicals.
CLAIMS

1. A method for the production of cellulose pulp in which a raw material, preferably cut wood chips, is treated in several stages at successively increasing temperatures, with at least one stage in which the raw material in at least one impregnation liquor is treated at a first impregnation temperature followed by cooking by the means of at least one cooking liquor, preferably an alkali cooking liquor, at a second cooking temperature, which cooking temperature lies within the interval 150±20°C, whereby the raw material undergoes several delignification stages characterized in that at least a fraction of the cooking liquor during one of the delignification stages is constituted by pre-evaporated black liquor, which is added to the raw material before the particular delignification stage, and that the pre-evaporated black liquor that is added before the cooking stage has a content of dry matter (TS) that exceeds the content of dry matter that can be obtained in the black liquor that is withdrawn from the process and subsequently being subjected to pressure reduction.

2. The method according to claim 1, characterized in that the pre-evaporated black liquor that is added prior to the cooking stage has a content of dry matter (TS) that lies at least 10% above the content of dry matter that can be obtained from the black liquor that is withdrawn from the process and subsequently being subjected to pressure reduction.

3. The method according to claim 2, characterized in that the black liquor that is added prior to the cooking stage has a content of dry matter (TS) that exceeds 30%, and that preferably exceeds 40%.

4. The method according to claim 3, characterized in that the black liquor that is added prior to the cooking stage has a content of dry matter (TS) that exceeds 50%.
5. The method according to claim 2, characterized in that the pre-evaporated black liquor is mixed with other treatment liquor before it is added to the cellulose material prior to it being cooked in the relevant delignification stage.

6. The method according to any one of the preceding claims, characterized in that the black liquor that has been obtained from the impregnation or cooking stage first has its pressure reduced in at least one pressure-reduction tank down to a pressure that does not exceed an excess pressure of 0.5 bar, whereafter the pressure-reduced black liquor is evaporated in at least one primary evaporation step in which the black liquor is heated during the removal of volatile substances from the black liquor, giving a black liquor obtained after the evaporation stage with a higher content of dry material.

7. The method according to claim 6, characterized in that the relevant primary evaporation step is part of a recovery plant for black liquor, in which the primary evaporation stages that are used to increase the content of dry material in the black liquor prior to its return to the impregnation or cooking stage are constituted by first stages in an evaporation line with several evaporation stages, and from which primary evaporation stages a part of the pre-evaporated black liquor from the complete amount of black liquor that has been treated in the primary evaporation stages is removed for return to the impregnation or cooking stage, and from which evaporation line is finally obtained a highly evaporated black liquor that is subsequently combusted in a soda recovery furnace for recovery of the alkali through obtaining a melt that is subsequently mixed to give green liquor.

8. The method according to claim 6, characterized in that the primary evaporation stage is constituted by a specially dedicated evaporation stage that only evaporates the amount of black liquor that is to be returned to the impregnation or cooking stage.
9. The method according to any one of the preceding claims, characterized in that the pre-evaporated black liquor is added to the cooking liquor whereby this pre-evaporated black liquor is present during at least part of the initial delignification stage of the cooking stage.

10. The method according to claim 9, characterized in that the pre-evaporated black liquor is added to the cooking liquor whereby this pre-evaporated black liquor is present during more than 50% of the bulk delignification stage of the cooking stage.
A. CLASSIFICATION OF SUBJECT MATTER

IPC7: D21C 3/02
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)

IPC7: D21C

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

SE,DK,FI,NO classes as above

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Further documents are listed in the continuation of Box C. See patent family annex.

Date of the actual completion of the international search: 14 April 2003

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### INTERNATIONAL SEARCH REPORT

**Information on patent family members**

**29/03/03**

**International application No.**

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