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(54) **IMPROVED COMPACT PROCESS FOR PRODUCING PREHYDROLYZED PULP**

VERBESSERTES KOMPAKTES VERFAHREN ZUR HERSTELLUNG VON VORHYDROLYSIERTEM ZELLSTOFF

PROCÉDÉ COMPACT AMÉLIORÉ POUR LA PRODUCTION D'UNE PÂTE PRÉHYDROLYSÉE

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Description

FIELD OF THE INVENTION

[0001] The present invention relates to a process for production of pulp in which hemicellulose is hydrolyzed into hydrolysate, and lignin is dissolved by a kraft cooking method for liberating cellulose fibers. Still more particularly, the present invention relates to a process for production of a pulp which has a high content of alpha cellulose and can be sold as dissolving pulp.

BACKGROUND OF THE INVENTION

[0002] Traditionally, there are basically two processes for production of special pulps having a high content of alpha cellulose. These include acidic sulfite cooking, and prehydrolysis- sulfate (kraft) cooking. The former was developed at the end of the 19th century, and the latter in the 1930's, see, e.g., Rydholm, S. E., Pulping Processes, pp. 649 to 672, Interscience Publishers, New York, 1968. The basic idea in both processes is to remove as much hemicellulose as possible from cellulose fibers in connection with delignification, so as to obtain a high content of alpha cellulose, i.e. polymeric chains having a relative high polymerization degree and not short hemicellulose molecules with a randomly grafted molecular structure. In the traditional sulfite process, the removal of hemicelluloses takes place during the cooking, simultaneous with dissolving of the lignin. The cooking conditions in that case are highly acidic, and the temperature varies from 140 °C to 150 °C, whereby hydrolysis is promoted. The result, however, is always a compromise with delignification. A drawback is the decrease in the degree of polymerization of the alpha cellulose and yield losses, which also limit the potential for hydrolysis. Various improvements have thus been suggested, such as modification of the cooking conditions, and even a prehydrolysis step followed by an alkaline sulfite cooking stage. The main obstacle in connection with sulfite pulping processes is the complicated and costly recovery processes of the cooking chemicals.

[0003] A separate prehydrolysis step permits the desired adjustment of the hydrolysis of hemicelluloses by varying the hydrolysis conditions. In the prehydrolysis-kraft cooking process, the bulk delignification is not carried out until a separate alkaline cooking step, even though some handbooks indicate that as much as 30 kg of lignin per ton of wood may be dissolved in the prehydrolysis (i.e. a small part of the total lignin content as 30 kg per ton of wood corresponds to some 3 % of the wood material). The conditions for prehydrolysis is most often established by heating in a hot steam phase or hot water liquid environment, where the natural wood acidity released will usually lower the pH down to about 3.5, most often referred to as autohydrolysis. Sometimes could also additional acid and a catalyst be added. The subsequent delignification step has been a conventional kraft cooking method, where white liquor has been added to the digester.

[0004] Several prehydrolysis-kraft cooking processes have been disclosed and this technology was very much in focus during the late 60-ies and early 70-ies. In the early publication "Continuous Pulping Processes" by Sven Rydholm, 1970, is described the experiences from "Continuous Prehydrolysis-kraft Pulping" on pages 105-119. On page 106, Fig. 8.1, is disclosed a two-vessel continuous cooking system with a first up-flow prehydrolysis tower followed by a downflow conventional kraft cooking digester, in which the up-flow tower experienced severe pitch deposits on the extraction strainers which clogged after only 3-6 days of operation. Another system is disclosed on page 107, Fig. 8.2, with a one vessel hydraulic continuous digester system with a first upper prehydrolysis zone and a lower alkaline kraft cooking zone, both zones being separated by a strainer section. However, also in this one vessel design were the strainers subjected to severe pitch deposits and clogging. The pitch problem also migrated into the chip feeding system resulting in the need for an alkali charge in the high-pressure feeder to avoid pitch deposition in the high-pressure feeder.

[0005] In US 8,734,610 is disclosed a two-vessel digester system, where the acidic hydrolysis is established in an upper half of a first prehydrolysis vessel, followed by a countercurrent water-wash stage with some alkali addition. This system is of similar design as the old Varkaus (Finland) digester system that had the inherent pitch problems in the screen section ending the hydrolysis zone, and this system could only be operated in short campaigns with dissolving pulp production, said campaign being maximized to about two weeks at the most, requiring a swing production to standard kraft pulping process after these two weeks. The old Varkaus system is described in detail in US 4,436,586 (1984).

[0006] This pitch problem has been seen in almost every installation of continuous cooking systems used for prehydrolysis-kraft pulping. This causes production disturbances and pulp quality variations. Furthermore, the lack of a well-defined prehydrolysis zone in previous continuous installations has caused variations in the degree of hydrolyzation, which in turn led to unacceptable quality variations of the final product.

[0007] When prehydrolysis-kraft pulping is implemented in batch systems, the pitch problem is partially solved by the fact that the strainers in the batch digester are switching from withdrawing acidic prehydrolysate to alkaline cooking liquor and later on black liquor. The volume of acidic hydrolysate withdrawn after a steam phase prehydrolysis is also relatively small in total volume so the exposure in strainers is limited. The latter alkaline stages will then also dissolve and wash out any pitch deposits such that they do not build up over time. This is not possible to achieve in continuous systems as the strainers are located in a stationary process position where the chemical conditions (as of pH, pitch content, etc.) do

not change.

[0008] In US 5,589,033 is disclosed a batch process for prehydrolyzed kraft pulp sold by Valmet, often in connection with Superbatch™ cooking. Here is a hot 170 °C prehydrolysis step in a gaseous steam phase terminated by a hot neutralization step at 155 °C using heated alkali and for a duration of only 15 minutes (as shown in Example 3). This neutralization is followed by a hot black liquor treatment step at 148 °C for a duration of 20 minutes, and finally the pulp is cooked in a kraft-cooking stage at 160 °C for 54 minutes. Here the degree of hydrolyzation could be controlled in a good manner by controlling the duration of each stage. As the hydrolysis step is conducted in a steam phase, can the following neutralization and alkalization of the wood material be obtained rather quickly and thoroughly as the wood material has been steamed at high temperature in a steam phase, allowing the alkali to penetrate the wood material by diffusion. However, this type of well-defined transition zone between the prehydrolyse in a steam phase and the neutralization is not favorable in a continuous system where the wood material is supposed to flow through reaction towers in a plug flow. Hence, the hydrolysis is instead most often implemented in a liquid filled stage at least in final parts.

[0009] Nowadays dissolving pulp for such end uses as spinning fibers (rayon/lyocell) is considered to be an optional method for producing textiles having less environmental impact compared with production of cotton textiles. The interest for dissolving pulp increases in years when cotton production is low due to crop failure. Dissolving pulp is also a base product for different additives and consistency agents and fillers in tyre cord and casings, ether and spongs, nitrocellulose and acetate. Hence, dissolving pulps may be an alternative product instead of pulp for regular paper pulp making.

[0010] A common implementation in most prehydrolysis-kraft cooking processes is that the prehydrolysis stage has been terminated by withdrawal of the prehydrolysate, either in form of a pure acidic prehydrolysate, or in form of a neutralized prehydrolysate. As indicated before would any strainers in such process position be subjected to pitch deposits, both when the prehydrolysate is kept at its lowest pH level or if the prehydrolysate is withdrawn in a transition position where the chip suspension switch from acidic to alkaline.

[0011] In WO 2012158075 (Metso Paper Sweden which now is Valmet AB) is disclosed an alternative compact process for producing dissolving pulp using a prehydrolysis kraft cooking process. In this process is a charge of colder alkali added to the hydrolysed cellulosic slurry containing all dissolved carbohydrates, which charge quickly terminates the process conditions for hydrolysis. In a first embodiment is a three-vessel design proposed, where an intermediate alkaline extraction process is established after the first hydrolysis vessel, enabling a further extraction of both hemicellulose and lignin from the hydrolysed cellulosic slurry. In a second embodiment is a two-vessel design proposed where the charge of colder alkali is added to the end of the first hydrolysis vessel, and a first withdrawal to recovery is made in a digester screen section.

[0012] US 2012/158075 A1 is related to an improved compact process for producing dissolving pulp in a prehydrolysis kraft cooking process. For avoiding pitch problems with blocked withdrawal screens and to obtain a distinct ending of the prehydrolysis stage, as well as a thorough alkaline impregnation ahead of the kraft cook stage, alkali is charged to the mixture of prehydrolyzed material to such an extent that the residual alkali concentration after neutralization of the acidic hydrolysate is above 20 g/l EA as NaOH and the temperature of the resulting alkaline treatment liquor for the prehydrolyzed material is lowered by at least 10% in comparison to the temperature in the prehydrolysis stage. The alkali charge will avoid redeposition of hemicelluloses dissolved in the prehydrolyse stage and will abruptly swing the wood material mixture to alkaline conditions favourable for a alkali impregnation stage at reduced temperature ahead of the final kraft cooking stage, which impregnation stage will extract the major part of the hemicelluloses content of the cellulose material.

[0013] US 2012/211183 A1 relates to dissolving pulp by cooking and particularly with pre-hydrolysis and kraft cooking of wood chips. The pulp cooking system includes a pre-hydrolysis vessel and transfer system having multiple extraction points to remove the products of hydrolysis as the products are formed in the vessel and transfer system.

SUMMARY OF THE INVENTION

[0014] With the experience from the process solutions suggested in WO 2012158075 is a further improvement and simplification of the prehydrolysis-kraft process developed. It has surprisingly been found that the neutralization process after the hydrolysis need not to be swung to extreme alkali concentration above 20 g/l, and it is not necessary to implement an intermediate vessel for extended alkali extraction.

[0015] The inventive concept is based on a moderate alkalizate ion of the hydrolysed cellulosic slurry and using mixing effects of process components in the transfer system to a subsequent digester before a first withdrawal of hydrolysis liquid in a top separator and within a very short time frame less than 10 minutes after alkali addition.

[0016] In accordance with the invention as defined in the present claim 1, these and other objectives have now been accomplished by a process for the preparation of pulp from lignin-containing cellulosic material, comprising prehydrolyzing said cellulosic material in a prehydrolysis stage at a temperature of between about 120 °C and 180 °C and during at least 20 minutes, so as to produce a prehydrolyzed cellulosic material and an acidic hydrolysate. Adding a neutralizing alkali charge to the mixture of prehydrolyzed cellulosic material and acidic hydroly-

sate to such extent that the alkali concentration directly after the alkali charge is between 5-10 g/l EA as NaOH thereby forming a neutralized slurry containing the hydrolysed cellulosic material and the hydrolysate, maintaining the neutralized slurry for a short time not exceeding 10 minutes in the neutralized state while subjecting the neutralized slurry by mechanical agitation, e.g. by at least one bottom scraper (BS), whereby dissolved carbohydrates as well as any lignin dissolved in the prehydrolysis stage are maintained dissolved during this neutralization stage. Thereafter transferring the neutralized slurry directly to a kraft cooking stage using a transfer circulation with a feeding line to the top of a kraft cooking digester having a top separator extracting neutralized treatment liquor, and sending extracted neutralized treatment liquor at least in part back to the start of the transfer circulation, and wherein at least 0.3 m^3/BDT of wood is withdrawn from the extracted neutralized treatment liquor and sent (e.g. directly) to recovery. Preferably is at least 0.3 m^3/BDT of wood is withdrawn, but as much as 0.5-2 m^3/BDT of wood may be withdrawn.

[0017] With this implementation are several advantages obtained, such as:

- Minimized charge of alkali and no risk for any larger order of alkali consumption during the short retention time in the transfer system (typically less than 2-3 minutes);
- Pitch deposits and clogging in top separator screen are prevented by thorough mixing effect of the added alkali by mechanical agitation, e.g. by a bottom scraper, a potential pump in transfer line, and/or the top separator screw; and
- The process can readily be implemented in a cost-effective investment in a simple two-vessel prehydrolysis kraft system.

[0018] In a preferred embodiment of the inventive process is the neutralized slurry maintained for a short time not exceeding 5 minutes, preferably in the range of 1-2 minutes, in the neutralized state while subjecting the neutralized slurry by mechanical agitation before extraction from the top separator.

[0019] According to a preferred embodiment of the inventive process, the neutralizing alkali charge can contain at least one of fresh white liquor, extracted black liquor from a digester, or alkaline wash liquor from subsequent digester or alkaline pre-bleaching.

[0020] As the order of alkalinity to be established is relatively modest, i.e. 5-10 g/l EA as NaOH, which is in level with conventional residual alkali levels in black liquor withdrawn from a digester, typically lying at about 8 g/l, and sent to recovery, can preferably withdrawn cooking or washing filtrates be used as neutralizing liquids, and the more expensive white liquor can be used at rather small charged volumes in the neutralization stage.

[0021] In yet a preferred embodiment, the neutralizing alkali charge can be added in such amount and at such temperature that the resulting temperature of the neutralized slurry is lowered by at least 10 % in comparison to the temperature in the prehydrolysis stage, preferably at least 12 °C if the prehydrolyse temperature is about 120 °C, and at least 18 °C if the prehydrolyse temperature is about 180 °C. A substantial reduction in temperature will effectively end the prehydrolysis of the cellulose material, in combination with the swing to moderate alkaline conditions, i.e. 5-10 g/l. Preferably the resulting temperature is the same as the temperature to be established in the subsequent digester, reducing need to add steam to digester top for heating.

[0022] If the neutralization stage is kept short, i.e. less than 10 minutes, this also implies that the alkali concentration established cannot be subjected to any larger order of alkali consumption during delignification reaction processes, which forms the very foundation for keeping the alkalization in the neutralization at a moderate level and not risking the concentration to drop so much that pitch deposits are formed on screens in subsequent withdrawal.

[0023] In a further embodiment, also additional alkaline liquors can be added to the feeding line ahead of top separator, said additional alkaline liquors added in an amount sufficient for establishing a L/W ratio sufficient for maintaining the withdrawal capacity in the top separator and a high extraction volume sent to recovery in return line from the top separator, thereby avoiding top separator plugging. It has been seen in several digesters that the top separator needs to be operated with a certain overflow of liquor, as high degree of drainage of liquid in the withdrawal compartment, lowering the liquid level, may drain the plug of cellulose material excessively, and thus may form a plug that may activate the overload stop in the drive of the top separator motor. Adding additional liquid to the feeding line may quickly control the liquid overflow of the top separator, and maintain the withdrawal capacity high.

[0024] Further, in still another embodiment, also the slurry after the addition of neutralizing alkali charge can be subjected to mechanical agitation first from a bottom scraper and finally from a top separator having a feeding screw sweeping over withdrawal screens. These components add a mixing effect into the neutralized slurry and guarantee that the alkali concentration is established evenly throughout the entire volume of the neutralized slurry, thus reducing pitch deposits forming on top separator screen. In order to add an additional mixing effect can also the slurry after the addition of neutralizing alkali charge be subjected to mechanical agitation from a pump located in the feeding line to the top of a kraft cooking digester. This pump can in some systems be discarded as the pressure in the outlet from the prehydrolyzing vessel often is sufficient for establishing a flow in the feeding line.

[0025] The inventive process can be implemented in any type of prehydrolysis stage, such as a one where the

acidification of said prehydrolysis is established only by steam heating and optionally adding water, and without adding any external acidifiers, only using the wood acidity released during steam heating reaching a pH level below 5 during the prehydrolysis. It can also be implemented in one where the acidification of said prehydrolysis is established in a liquid-filled phase wherein the acidification of said prehydrolysis is established by heating and addition of external acidifiers, reaching a pH level below 3 during the prehydrolysis established in a liquid-filled phase.

[0026] Finally, the inventive process can be implemented in a continuous digester system using one vessel for the prehydrolysis and one vessel for an alkaline pre-extraction stage and the kraft cooking stage.

[0027] The lignin-containing cellulosic materials to be used in the present process are suitably softwood, hardwood, or annual plants. According to the present invention, prehydrolysis-kraft pulp can be obtained with a high yield of alpha cellulose with a high polymerization degree.

BRIEF DESCRIPTION OF THE DRAWINGS

[0028]

Fig. 1 is a schematic representation of the prehydrolysis-kraft cooking process according to US 5,589,033;

Fig. 2 is a schematic representation of the of the prehydrolysis-kraft cooking process according to WO 2012158075;

Fig. 3 is a schematic representation of the cooking process according to the invention; and

Fig. 4 shows a principal set up for a continuous cooking system using the inventive process, here using a prehydrolysis tower and one subsequent vessels for alkaline treatment and cook.

DETAILED DESCRIPTION OF THE INVENTION

[0029] As a comparison is in Fig. 1 shown the cooking steps of US 5,589,033. The chips are first treated in the prehydrolysis step Pr, where chips are heated by steam to 170 °C for 25 minutes. Thereafter is heated white liquor added in order to establish a neutralization step Ne, and the acidic prehydrolysate RECAc is withdrawn from the process. The neutralization step is established at 155 °C for 15 minutes. Even though the white liquor is heated, the temperature is decreased by some 8 %. After the neutralization step, the neutralization liquid is displaced by adding hot black liquor BL_{HOT}, and this establishes an alkaline black liquor impregnation step BL held at 148 °C for 20 minutes. Thereafter the black liquor is withdrawn, and a new charge of white liquor is added ahead of the following cooking step Co, which is held at 160 °C for 54 minutes. In commercial batch cooking systems like Superbatch™ sold by Valmet, the white liquor used is heated both in a heat exchange with hot spent cooking liquor as well as steam in order not keep the temperature at high level, before being used as the neutralizing liquid.

[0030] Fig. 2 shows the cooking steps of WO 2012158075. Here is shown a first steaming step ST for the chips but this step may be avoided of the subsequent prehydrolysis is implemented in a steam phase. The chips are thereafter treated in the prehydrolysis step Pr, where chips are heated by steam at a temperature of between about 120 °C and 180 °C and during at least 20 minutes, to produce a prehydrolyzed cellulosic material and an acidic hydrolysate. Addition of liquid, such as water H_2O, is an option, which may be preferable if a liquid prehydrolysis is sought for, for example in a continuous cooking system. Another option is to add an acidifier Ac, if a lower temperature is sought for in the prehydrolysis.

[0031] In Fig. 3 are instead the basic process steps of the present invention shown. As in Fig. 2 is shown a first steaming step ST for the chips but this step can be avoided if the subsequent prehydrolysis is implemented in a steam phase. The chips are thereafter treated in the prehydrolysis step Pr, where chips are heated by steam at a temperature of between about 120 °Cand 180 °C and during at least 20 minutes, to produce a prehydrolyzed cellulosic material and an acidic hydrolysate. Addition of liquid, such as water H_2O, is an option, which can be preferable if a liquid prehydrolysis is sought for, for example in a continuous cooking system. Another option is to add an acidifier Ac if a lower temperature is sought for in the prehydrolysis. The novel approach disclosed in this figure is the usage of the transfer circulation Tr as the position for first withdrawal of dissolved matter from the hydrolysis stage. The residence time for the hydrolysed slurry in the transfer circulation is typically within 1-3 minutes, and well below 10 minutes. Directly ahead of the transfer circulation is a moderate alkali charge AL added to the hydrolysed slurry. According to the inventive process is a distinct ending of the prehydrolysis implemented by adding a moderate alkali charge AL with a volume and at a temperature that will reduce the temperature of the cellulosic material by at least 10 % in comparison to the temperature in the prehydrolysis stage, preferably at least 12 °C if the prehydrolyse temperature is about 120 °C and at least 18 °C if the prehydrolyse temperature is about 180 °C. This will establish an alkaline treatment liquor which after this charge establishes an effective alkali concentration in the range 5-10 g/l EA as NaOH. Thereafter the neutralized slurry is withdrawn within a short period of time less than 10 minutes and preferably within 1-3 minutes while having been subjected to mechanical mixing effects from at least a bottom scraper and a top separator screw.

[0032] The short retention time in the transfer system Tr guarantees that no major consumption of alkali is established, which is the basis for keeping the charge of alkali at relatively modest level. The charge of fresh alkali, i.e. typically in form of concentrated white liquor

directly from the white liquor preparation in the causticizing area can thus be reduced, and to some extent be replaced by alkaline filtrates from brown wash (wash directly after digester or after a first oxygen delignification stage) or black liquor extraction flows from digester.

[0033] Thereafter the cellulosic material is transferred from the transfer system to a kraft cooking stage Co. The kraft cook can be implemented in any kind of known kraft cooking method continuous cooking such as, Compact Cooking, Lo-Solids cooking, ITC-cooking, MCC cooking, EAPC cooking as examples. The kraft cook is then finished by a wash stage Wa, which can be implemented in any kind of known wash equipment, such as a counter-current wash zone in bottom of a digester or using a pressure diffuser 40 wash, wash press or filter wash after the cook.

[0034] In Fig. 4 is disclosed a two-vessel continuous cooking system for prehydrolysis and cooking, wherein the inventive process is implemented. The chips are first fed to a chip bin 1 and subsequent steaming vessel 2 during addition of steam ST for purging the chips from bound air. From the steaming vessel, the steamed chips fall into a liquid-filled chute above a high-pressure sluice feeder 3, which pressurize the steamed chips and feed the formed slurry of chips in a feed flow 4 to the prehydrolysis vessel 10. Here the prehydrolysis vessel is in form of a steam-liquid phase digester having an inverted top separator 11 withdrawing a part of the transport liquid from line 4 back to start of feeding via A. As indicated, steam ST is added to top of vessel 10, and optionally can also acid be added from source Ac. In bottom of the prehydrolysis vessel 10 is the moderate alkali charge added from any suitable source, such as extracted black liquor withdrawals from digester, REC2 and REC4, as well as alkaline wash filtrate (REC3) from a pressure diffuser wash 40 located directly after the digester. The addition of the moderate alkali charge is done by mixing in the alkali to the return flow B. The neutralized slurry is fed in line 14 to a steam-liquid phase digester 30, and excess transport fluid is withdrawn by an inverted top separator 31 and sent to B, which is added to bottom of the prehydrolysis vessel 10 as part of the transfer circulation. From the return flow a substantial part of the neutralizing liquor can be withdrawn. The kraft cook is established in the digester 30 and finally the prehydrolysed and cooked pulp is washed in a pressure diffuser 40.

Claims

1. A process for the preparation of pulp from lignin-containing cellulosic material, comprising prehydrolyzing said cellulosic material in a prehydrolysis stage at a temperature of between 120 °C and 180 °C and during at least 20 minutes so as to produce a prehydrolyzed cellulosic material and an acidic hydrolysate, **characterized by** adding a neutralizing alkali charge to the prehydrolyzed cellulosic material and acidic hydrolysate to such extent that the alkali concentration directly after the alkali charge is between 5-10 g/l EA as NaOH thereby forming an neutralized slurry containing the hydrolysed cellulosic material and the hydrolysate, maintaining the neutralized slurry for a short time not exceeding 10 minutes in the neutralized state while subjecting the neutralized slurry by mechanical agitation, whereby dissolved carbohydrates as well as any lignin dissolved in the prehydrolysis stage are maintained dissolved during this neutralization stage, thereafter transferring the neutralized slurry directly to a kraft cooking stage using a transfer circulation with a feeding line to the top of a kraft cooking digester having a top separator extracting neutralized treatment liquor and sending extracted neutralized treatment liquor at least in part back to the start of the transfer circulation, and wherein at least 0.3 m^3/BDT of wood is withdrawn from the extracted neutralized treatment liquor and sent to recovery.

2. The process of claim 1, **characterized by** that the neutralized slurry is maintained for a short time not exceeding 5 minutes, preferably in the range 1-2 minutes, in the neutralized state while subjecting the neutralized slurry by mechanical agitation before extraction from the top separator.

3. The process of claim 1, **characterized by** that the neutralizing alkali charge contains at least one of fresh White liquor (WL), extracted black liquor from a digester (REC2, REC4), or alkaline wash liquor (REC3) from subsequent digester or alkaline pre-bleaching.

4. The process of claim 3, **characterized by** that the neutralizing alkali charge is added in such amount and at such temperature that the resulting temperature of the neutralized slurry is lowered by at least 10 % in comparison to the temperature in the prehydrolysis stage, preferably at least 12 °C if the prehydrolyse temperature is 120 °C and at least 18 °C if the prehydrolyse temperature is 180 °C.

5. The process of claim 4, **characterized by** that the resulting temperature is the same as the temperature to be established in the subsequent digester, reducing need to add steam to digester top for heating.

6. The process of claim 1, **characterized by** that additional alkaline liquors is added to the return line from the top separator, said additional alkaline liquors added in an amount sufficient for establishing a L/W ratio over the top separator of at least 2.0 m^3/Bdt wood even at high extraction flows from the transfer return line to avoid top separator plugging.

7. The process of claim 1, **characterized by** that the slurry after the addition of neutralizing alkali charge is subjected to mechanical agitation from a bottom scraper (BS).

8. The process of claim 7, **characterized by** that the slurry after the addition of neutralizing alkali charge is subjected to mechanical agitation first from a bottom scraper (BS) and finally from a top separator (31) having a feeding screw sweeping over withdrawal screens.

9. The process of claim 8, **characterized by** that the slurry after the addition of neutralizing alkali charge is subjected also to mechanical agitation from a pump (P) located in the feeding line (14) to the top of a kraft cooking digester (30).

10. The process of claim 1, **characterized by** that the acidification of said prehydrolysis is established only by steam heating and optionally adding water, and without adding any external acidifiers, only using the wood acidity released during steam heating reaching a pH level below 5 during the prehydrolysis.

11. The process of claim 1, **characterized by** that the acidification of said prehydrolysis is established in a liquid filled phase, wherein the acidification of said prehydrolysis is established by heating and addition of external acidifiers, reaching a pH level below 3 during the prehydrolysis established in a liquid filled phase.

12. The process of claim 1, **characterized by** that the process is implemented in a continuous digester system using one vessel (10) for the prehydrolysis and one vessel (30) for an alkaline pre-extraction stage and the kraft cooking stage.

Patentansprüche

1. Verfahren zur Herstellung von Zellstoff aus ligninhaltigem Cellulosematerial, umfassend Vorhydrolysieren des Cellulosematerials in einer Vorhydrolysestufe bei einer Temperatur zwischen etwa 120 °C und 180 °C und während wenigstens 20 Minuten, um ein vorhydrolysiertes Cellulosematerial und ein saures Hydrolysat zu erzeugen, **gekennzeichnet durch** Zugabe einer neutralisierenden Alkaliladung zu dem vorhydrolysierten Cellulosematerial und dem sauren Hydrolysat in einem solchen Ausmaß, dass die Alkalikonzentration direkt nach der Alkaliladung zwischen 5 und 10 g/l EA als NaOH liegt, wodurch eine neutralisierte Aufschlämmung gebildet wird, die das hydrolysierte Cellulosematerial und das Hydrolysat enthält, wobei die neutralisierte Aufschlämmung für eine kurze Zeit von nicht mehr als 10 Minuten in dem neutralisierten Zustand gehalten wird, während die neutralisierte Aufschlämmung mechanischem Rühren unterzogen wird, wodurch gelöste Kohlenhydrate sowie jegliches in der Vorhydrolyse-Stufe gelöstes Lignin während dieser Neutralisationsstufe gelöst bleiben, anschließendes Überführen der neutralisierten Aufschlämmung direkt in eine Kraft-Kochstufe unter Verwendung eines Transferkreislaufs mit einer Zuführungsleitung zu dem oberen Teil eines Kraftkochers mit einem oberen Separator, der neutralisierte Behandlungsflüssigkeit extrahiert und extrahierte neutralisierte Behandlungsflüssigkeit wenigstens zum Teil zurück zu dem Anfang des Transferkreislaufs schickt, und wobei wenigstens 0,3 m^3/BDT Holz aus der extrahierten neutralisierten Behandlungsflüssigkeit entnommen und zur Rückgewinnung geschickt werden.

2. Verfahren gemäß Anspruch 1, **dadurch gekennzeichnet, dass** die neutralisierte Aufschlämmung für eine kurze Zeit, die 5 Minuten nicht überschreitet, vorzugsweise in dem Bereich von 1 bis 2 Minuten, in dem neutralisierten Zustand gehalten wird, während die neutralisierte Aufschlämmung mechanischem Rühren unterzogen wird, bevor sie aus dem oberen Abscheider extrahiert wird.

3. Verfahren gemäß Anspruch 1, **dadurch gekennzeichnet, dass** die neutralisierende Alkaliladung wenigstens eine von frischer Weißlauge (WL), extrahierter Schwarzlauge aus einem Kocher (REC2, REC4) oder alkalischer Waschlauge (REC3) aus einem nachfolgenden Kocher oder einer alkalischen Vorbleiche enthält.

4. Verfahren gemäß Anspruch 3, **dadurch gekennzeichnet, dass** die neutralisierende Alkaliladung in einer solchen Menge und bei einer solchen Temperatur zugegeben wird, dass die resultierende Temperatur der neutralisierten Aufschlämmung um wenigstens 10 % im Vergleich zu der Temperatur in der Vorhydrolyse-Stufe, vorzugsweise wenigstens 12 °C, , wenn die Vorhydrolyse-Temperatur etwa 120 °C beträgt, und wenigstens 18 °C erniedrigt wird, wenn die Vorhydrolyse-Temperatur etwa 180 °C beträgt.

5. Verfahren gemäß Anspruch 4, **dadurch gekennzeichnet, dass** die resultierende Temperatur dieselbe Temperatur ist, wie die, die in dem nachfolgenden Kocher hergestellt werden soll, wodurch die Notwendigkeit verringert wird, Dampf zum Erhitzen oben in den Kocher zuzugeben.

6. Verfahren gemäß Anspruch 1, **gekennzeichnet durch** die Zugabe von zusätzlichen alkalischen Laugen in die Rückführleitung von dem oberen Abscheider, wobei die zusätzlichen alkalischen Laugen in

einer Menge zugegeben werden, die ausreicht, um ein L/W-Verhältnis über dem oberen Abscheider von wenigstens 2,0 m^3/Bdt Holz selbst bei hohen Extraktionsströmen aus der Transferrückführleitung zu erreichen, um ein Verstopfen des oberen Abscheiders zu vermeiden.

7. Verfahren gemäß Anspruch 1, **dadurch gekennzeichnet, dass** die Aufschlämmung nach der Zugabe der neutralisierenden Alkaliladung einem mechanischen Rühren durch einen Bodenschaber (BS) unterzogen wird.

8. Verfahren gemäß Anspruch 7, **dadurch gekennzeichnet, dass** die Aufschlämmung nach der Zugabe der neutralisierenden Alkaliladung zuerst einem mechanischen Rühren durch einen Bodenschaber (BS) und schließlich von einen oberen Abscheider (31) mit einer Zuführschraube, die über Entnahmesiebe streicht, unterzogen wird.

9. Verfahren gemäß Anspruch 8, **dadurch gekennzeichnet, dass** die Aufschlämmung nach der Zugabe der neutralisierenden Alkaliladung auch einem mechanischen Rühren durch eine Pumpe (P) unterzogen wird, die sich in der Zufiihrleitung (14) zu der Spitze eines KraftKochers (30) befindet.

10. Verfahren gemäß Anspruch 1, **dadurch gekennzeichnet, dass** die Ansäuerung der Vorhydrolyse nur durch Dampfheizung und gegebenenfalls Zugabe von Wasser und ohne Zugabe von externen Säuerungsmitteln erfolgt, wobei nur die während der Dampfheizung freigesetzte Holzsäure verwendet wird, die während der Vorhydrolyse einen pH-Wert unter 5 erreicht.

11. Verfahren gemäß Anspruch 1, **dadurch gekennzeichnet, dass** die Ansäuerung der Vorhydrolyse in einer flüssigkeitsgefüllten Phase erfolgt, wobei die Ansäuerung der Vorhydrolyse durch Erhitzen und Zugabe externer Säuerungsmittel erfolgt, wobei während der Vorhydrolyse, die in einer flüssigkeitsgefüllten Phase erfolgt, ein pH-Wert unter 3 erreicht wird.

12. Verfahren gemäß Anspruch 1, **dadurch gekennzeichnet, dass** das Verfahren in einem kontinuierlichen Kochersystem durchgeführt wird unter Verwendung eines Behälters (10) für die Vorhydrolyse und eines Behälters (30) für eine alkalische Vorextraktionsstufe und die Kraft-Kochstufe.

Revendications

1. Procédé de préparation de pâte à papier à partir de matières cellulosiques contenant de la lignine, comprenant la préhydrolyse de ladite matière cellulosique dans une étape de préhydrolyse à une température comprise entre 120 °C et 180 °C et pendant au moins 20 minutes de manière à produire une matière cellulosique préhydrolysée et un hydrolysat acide, **caractérisé par** l'ajout d'une charge alcaline neutralisante à la matière cellulosique préhydrolysée et à l'hydrolysat acide de telle sorte que la concentration alcaline directement après la charge alcaline soit comprise entre 5 et 10 g/l EA sous forme de NaOH, en formant ainsi une suspension neutralisée contenant la matière cellulosique hydrolysée et l'hydrolysat, en maintenant la suspension neutralisée pendant une courte période ne dépassant pas 10 minutes à l'état neutralisé tout en soumettant la suspension neutralisée par agitation mécanique, tandis que les glucides dissous ainsi que toute lignine dissoute dans l'étape de préhydrolyse sont maintenus dissous pendant cette étape de neutralisation, puis en transférant la suspension neutralisée directement à une étape de cuisson sous pression au moyen d'une circulation de transfert avec une conduite d'alimentation vers la partie supérieure d'un digesteur de cuisson sous pression ayant un séparateur supérieur extrayant la liqueur de traitement neutralisée et renvoyant la liqueur de traitement neutralisée extraite au moins en partie au début de la circulation de transfert, et au moins 0,3 m^3/BDT de bois étant prélevé dans la liqueur de traitement neutralisée extraite et envoyé à la récupération.

2. Procédé selon la revendication 1, **caractérisé en ce que** la suspension neutralisée est maintenue pendant une courte durée n'excédant pas 5 minutes, de préférence dans la plage de 1 à 2 minutes, à l'état neutralisé tout en soumettant la suspension neutralisée par agitation mécanique avant extraction est du séparateur supérieur.

3. Procédé selon la revendication 1, **caractérisé en ce que** la charge alcaline neutralisante contient au moins une solution parmi la liqueur blanche fraîche (WL), la liqueur noire extraite d'un digesteur (REC2, REC4) ou la liqueur de lavage alcaline (REC3) d'un digesteur suivant ou d'un préblanchiment alcalin.

4. Procédé selon la revendication 3, **caractérisé en ce que** la charge alcaline neutralisante est ajoutée en une quantité et à une température telles que la température résultante de la suspension neutralisée soit abaissée d'au moins 10 % par rapport à la température dans l'étape de préhydrolyse, de préférence d'au moins 12°C si la température de préhydrolyse est de 120°C et d'au moins 18°C si la température de préhydrolyse est de 180°C.

5. Procédé selon la revendication 4, **caractérisé en ce**

que la température résultante est la même que la température à établir dans le digesteur suivant, ce qui réduit le besoin d'ajouter de la vapeur au sommet du digesteur pour le chauffage.

6. Procédé selon la revendication 1, **caractérisé en ce que** des liqueurs alcalines supplémentaires sont ajoutées dans la conduite de retour à partir du séparateur supérieur, lesdites liqueurs alcalines supplémentaires étant ajoutées en une quantité suffisante pour établir un rapport L/W au-dessus du séparateur supérieur d'au moins 2,0 m^3/Bdt de bois même à des débits d'extraction élevés à partir de la conduite de retour de transfert afin d'éviter un colmatage du séparateur supérieur.

7. Procédé selon la revendication 1, **caractérisé en ce que** la suspension, après l'ajout de la charge alcaline neutralisante, est soumise à une agitation mécanique par un racleur de fond (BS).

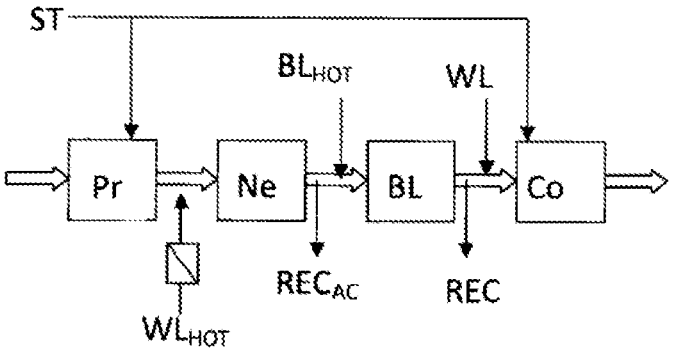
8. Procédé selon la revendication 7, **caractérisé en ce que** la suspension, après l'ajout de la charge alcaline neutralisante, est soumise à une agitation mécanique d'abord par un racleur de fond (BS) et par séparateur supérieur (31) ayant une vis d'alimentation balayant les tamis de prélèvement.

9. Procédé selon la revendication 8, **caractérisé en ce que** la suspension, après l'ajout de la charge alcaline neutralisante, est également soumise à une agitation mécanique d'une pompe (P) située dans la conduite d'alimentation (14) au sommet d'un digesteur de cuisson sous pression (30).

10. Procédé selon la revendication 1, **caractérisé en ce que** l'acidification de ladite préhydrolyse est établie uniquement par chauffage à la vapeur d'eau et éventuellement par ajout d'eau, et sans ajout d'acidifiants externes, uniquement en utilisant l'acidité du bois libérée pendant le chauffage à la vapeur d'eau atteignant un niveau de pH inférieur à 5 pendant la préhydrolyse.

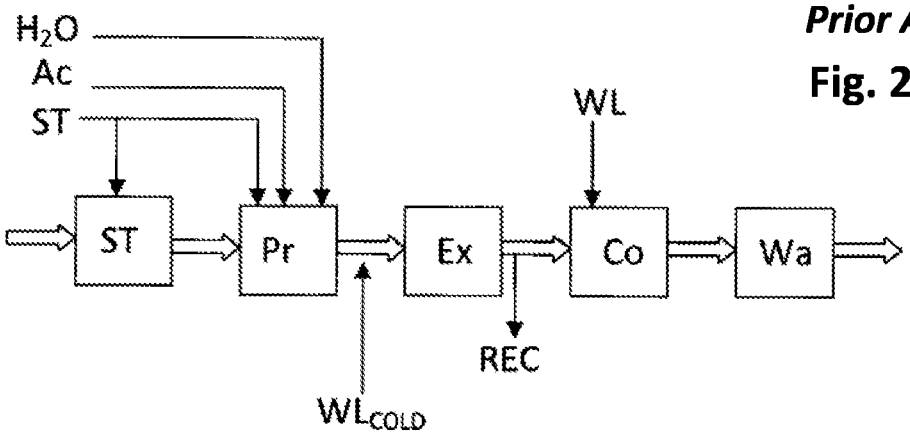
11. Procédé selon la revendication 1, **caractérisé en ce que** l'acidification de ladite préhydrolyse est établie dans une phase remplie de liquide, l'acidification de ladite préhydrolyse étant établie par chauffage et ajout d'acidifiants externes, en atteignant un niveau de pH inférieur à 3 pendant la préhydrolyse établie dans une phase remplie de liquide.

12. Procédé selon la revendication 1, **caractérisé en ce que** le procédé est mis en œuvre dans un système de digesteur continu en utilisant une cuve (10) pour la préhydrolyse et une cuve (30) pour une étape de pré-extraction alcaline et l'étape de cuisson sous pression.



Prior Art

Fig. 1



Prior Art

Fig. 2

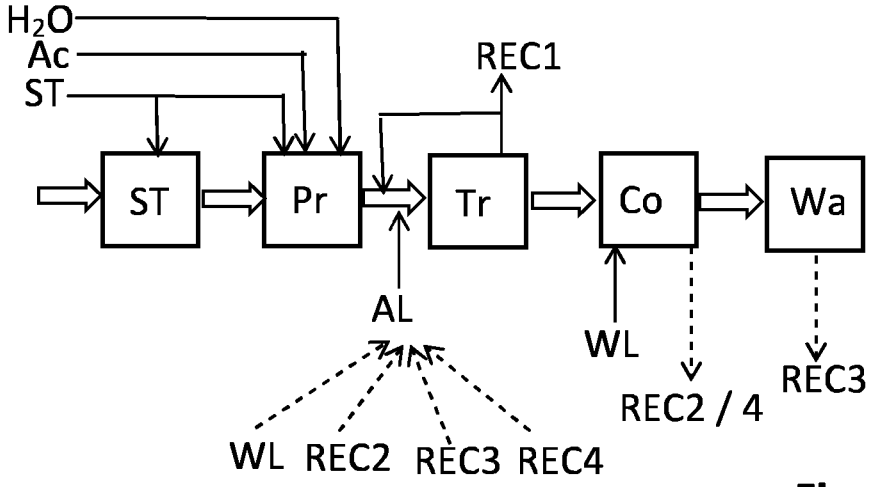


Fig. 3

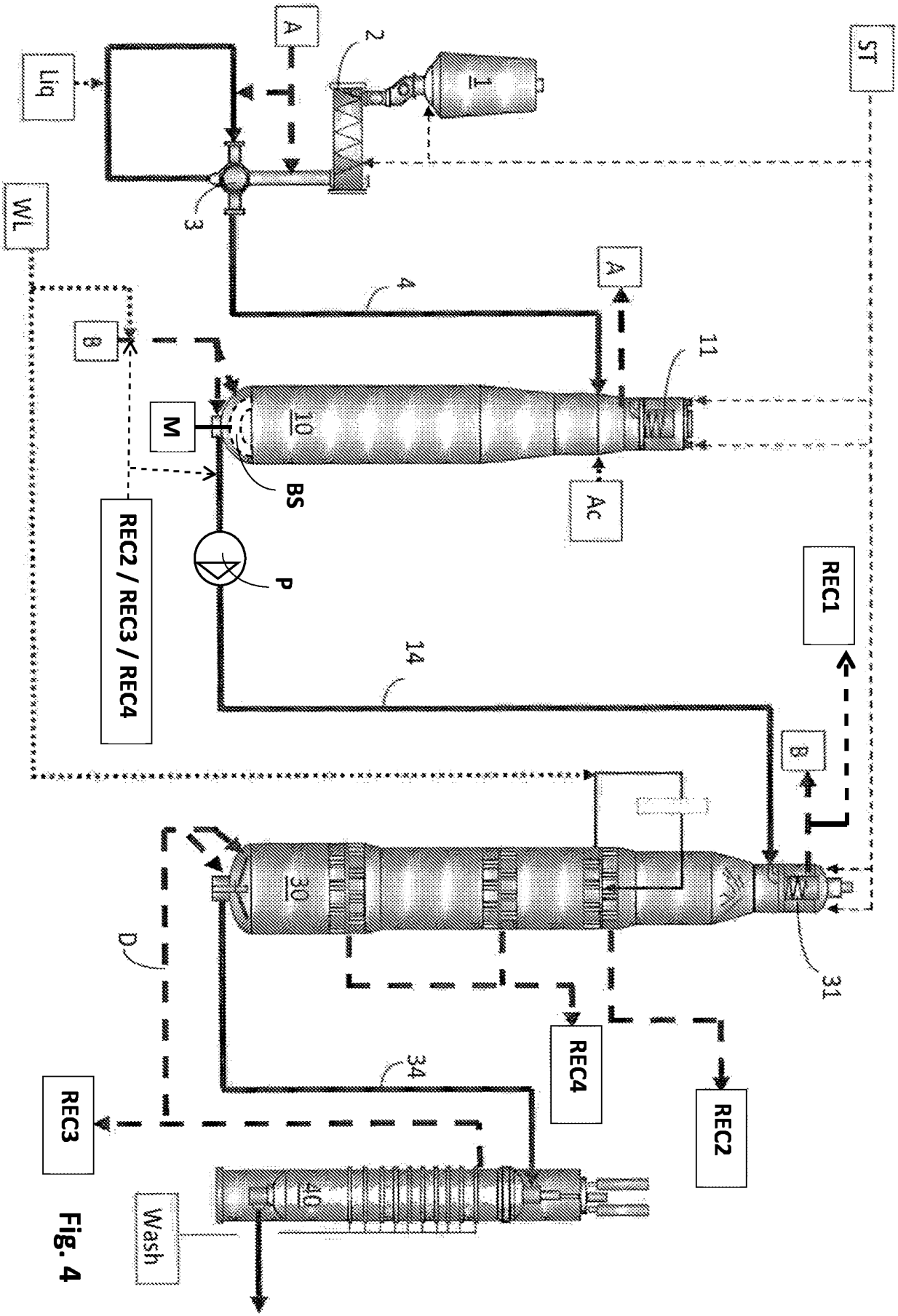

ST
Liq
A
2
1
9
WL
3
4
A
B
11
M
10
BS
Ac
REC2 / REC3 / REC4
P
REC1
14
B
30
31
D
REC4
REC2
34
REC3
40
Wash

Fig. 4

REFERENCES CITED IN THE DESCRIPTION

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