A method for producing pulp is disclosed. More particularly, the invention concerns a displacement batch cooking process comprising a steam-phase prehydrolysis step, wherein the recovery of the by-products is improved. In the recovery step of the present invention, after the target P-factor in the prehydrolysis stage is reached, hot washing liquid is introduced into the digester from the bottom thereof. The washing liquid is circulated via the suction screens to the top and to the bottom of the digester until the prehydrolyzed chips are under the washing liquid. The hot washing liquid containing by-products is recovered from the digester and the digester contents are neutralized by displacing the washing liquid with alkaline liquor.
METHOD FOR PRODUCING PULP

Field of the invention
The present invention relates to a method for producing pulp. More particularly, it concerns a displacement batch cooking process comprising a steam-phase prehydrolysis step, wherein the recovery of the by-products is improved.

Background of the invention
The prehydrolysis- sulfate (Kraft) cooking for the production of special pulps having a high content of alpha cellulose was developed in the 1930's, see e.g. Rydholm, S. E., Pulping Processes, pp. 649 to 672, Interscience Publishers, New York, 1968. The basic idea 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. This is essential because the various end uses of such pulps, dissolving pulp for instance, do not tolerate short-chained hemicellulose molecules with a randomly grafted molecular structure.

A separate prehydrolysis step permits the desired adjustment of the hydrolysis of hemicellulosic by varying the hydrolysis conditions. In the prehydrolysis-kraft cooking process the necessary delignification is not carried out until a separate second cooking step. The prehydrolysis is carried out either as a water or steam phase prehydrolysis, or in the presence of a catalyst. In the former processes, organic acids liberated from wood during the process perform a major part of the hydrolysis, whereas in the latter process, small amounts of mineral acid or sulfur dioxide may be added to "assist" the prehydrolysis. In the prehydrolysis stage carried out in a steam phase, direct steam is introduced to the chip column in the digester.

Traditionally after prehydrolyzing the cellulosic material in a reactor, the hydrolysate and the prehydrolyzed cellulosic material are neutralized in the reactor with alkaline neutralizing liquor so as to produce neutralized hydrolysate and neutralized prehydrolyzed cellulosic material. There is hydrolysate both in the free liquid outside the chips and also trapped and immobilized inside the chips. If desired, as much as possible of the hydrolysate can be recovered before the neutralization step in order to be able to
utilize the carbohydrates released in the prehydrolysis. A separate washing stage, in which the digester is first filled up with a washing liquid and then the liquid containing the carbohydrates is removed from the digester, can be used between the prehydrolysis and cooking stages. This is although time-consuming, unfavourable to the energy balance and produces a very dilute carbohydrate solution.

WO 2007/090925 describes an improved method for treating lignocellulosic material, wherein the digester and its contents are first heated with direct steam to a predetermined hydrolysis temperature and then a volume of washing liquid is introduced into the digester from one end and which washing liquid is removed from the other end of the digester, which is opposite to the introduction end.

According to the process described in patent publication WO 2007/090926 the hydrolysate is recovered by utilizing trickle-bed type down-flow of hydrolysate. In this method the first fraction of the trickled-down hydrolysate is collected as a product fraction and the second fraction is discharged from the digester to a hot hydrolysate storage tank to be used as the first trickle flow liquid in the next batch. By the trickle-bed type recovery it is obtained concentrated hydrolysate, but the recovery step is too slow and therefore it is disadvantageous to the pulp quality. Additionally, the treatment is uneven to the contents of the digester; it is obvious that channeling will occur during the treatment, the liquid goes there where it is easiest.

After the removal of the hydrolysate the process may continue by a neutralization-cooking process known in the art.

The object of the present invention is to optimize the recovery of the hydrolysate in a prehydrolysis sulfate cooking process. By the present method the disadvantages of the prior processes can be overcome or at least diminished remarkably. An even treatment to the whole contents of the digester is obtained, a high-grade dissolving pulp or paper grade pulp is obtained and the recovered hydrolysate can be obtained essentially pure of sulfur. Essentially pure of sulfur means that according to the invention it is possible that no sulfur containing liquids are added to the recovered hydrolysate i.e. according to one embodiment only sulfur originating from the wood is present. The further
processing of the carbohydrates is thus easier. Also the energy-efficiency of the process is taken into account.

Summary of the invention

The displacement batch pulping process for producing pulp according to the present invention comprises the following steps; chip filling, heating of the chips, prehydrolyzing of the chips, recovering of the hydrolysate including neutralizing of the digester contents, preparing the digester contents for kraft cooking by increasing the pH i.e. by adding alkaline cooking liquid and cooking to a required H-factor. In the recovery step of the present invention, after the target P-factor in the prehydrolysis stage is reached, hot washing liquid is introduced into the digester from the bottom thereof. The washing liquid can be hot water from the hot water accumulator. It is possible to add sodium hydroxide to the hot water, before introducing it into the digester, if needed. The digester circulation pump is started, as the level of the hot washing liquid in the digester is over the suction screens and the circulation to the top and the bottom of the digester is started. The pumping of the hot washing liquid is continued until all the chips are "under" the washing liquid. This recovery step washes the hydrolysate and the steam condensate produced during the steaming phase (i.e. prehydrolyzing step) from the chips to the washing liquid in order to have as high as possible concentration of the dissolved sugars/carbohydrates in the used washing liquid prior displacing it to the condensate accumulator. At the same time the prehydrolysis is stopped in a controlled way and thus the loss of fiber quality is prevented. The condensate i.e. washing liquid containing the by-products is displaced from the top of the digester with an alkaline liquid, the displacement of the condensate is the final part of the recovery step. The pH of the digester contents after the recovery step is finalized can vary between pH 9 - 12.

In other words the process according to the present invention for recovering by-products in a displacement batch pulping process in a digester comprising a bottom, a top and suction screens said method comprising prehydrolyzing of chips in a steam phase to produce hydrolysate and steam condensate wherein after the prehydrolysis stage the process continues by,

- introducing hot washing liquid into the digester
while adding the washing liquid, circulating it via the suction screens to the top and to the bottom of the digester
continuing the circulation until the prehydrolyzed chips are under the washing liquid
recovering the hot washing liquid containing by-products from the digester and neutralizing the digester contents by displacing the washing liquid with alkaline liquor.

Brief description of the figures
Fig. 1 is a schematic flow chart of the process according to one embodiment of the present invention, wherein hot water from HOT WATER accumulator is used as the washing liquid

Detailed description of the invention
The displacement batch pulping process according to the invention is started by filling the digester with the lignocellulosic material i.e. with the chips. The chip flow comes on a chip belt to the digester plant and from there to the digester(s) using chip screw conveyors on top of the digester(s). Low pressure (LP) steam is used to ensure good chip packing over the whole digester cross-section. During the chip filling, air is evacuated through the suction screens. Chip filling is stopped after the digester level switch has operated and the capping valve is then closed.

Heating of the chips is started by using low pressure (LP) steam from the top and bottom of the digester and the heating is continued with medium pressure (MP) steam, until the required temperature is reached i.e. 150 - 170 °C. The digester is kept at this temperature and pressure until the prehydrolysis step is completed, i.e. the required P-factor is reached. P-factor is a defined factor to control the prehydrolysis stage, taking the temperature and time into account (as e.g. H-factor); Herbert Sixta, Handbook of Pulp, Volume 1, Wiley-VCH Verlag, 2006, pages 343-345.

According to the invention the prehydrolysis step is carried out in a steam phase, where the acids of the wood are hydrolyzed by the steam and acidic conditions are created in the digester. The end-pH of the steam prehydrolysis stage varies depending
on the wood species and the prehydrolysis conditions itself. The pH is typically measured from the out coming liquid during the hot washing liquid fill and varies from 2.5 to 4.0.

After the P-factor is reached, the recovery step is started by introducing hot washing liquid to the digester. The temperature of the hot washing liquid is between 100 - 170°C, preferably between 140 - 160°C and it is pumped into the digester from the bottom thereof. According to one embodiment hot water from HOT WATER accumulator as shown in Fig. is used as the washing liquid. According to another embodiment sodium hydroxide is added to the hot water prior introducing it into the digester, if there is a need to adjust the pH of the recovery step to enhance the stopping of the prehydrolysis. When the washing liquid level reaches the suction screens, the digester circulation pump is started and the circulation to the top and bottom of the digester is started. The suction screens are normally located on the lower half of the digester, typically about on the height of one third from the digester bottom. The pumping of the washing liquid is continued until the level of the washing liquid reaches the level of the chips, i.e. all the chips are under the washing liquid. Typically this means that the washing liquid is added in an amount of about 40 to 80 % of the digester free volume. During the recovery step the hydrolysate and steam condensate produced during the prehydrolysis stage are washed from the chips to the washing liquid in order to have as high as possible concentration of the dissolved carbohydrates in the used washing liquid prior displacing it to the condensate accumulator. Additionally, the prehydrolysis stage is stopped during the recovery step in a controlled way in order to prevent the loss of fiber quality (e.g. viscosity and kappa number). The digester contents are thus neutralized. If hot water without addition of sodium hydroxide is used as the washing liquid, the pH of the contents of the digester before the displacement of the condensate varies between pH 5 - 7. On the other hand if the washing liquid contains alkali the pH of the digester contents before the displacement is typically pH 7 or more.

The final neutralization of the digester contents if any is achieved during the displacement of the condensate. The displacement of the condensate is started by stopping the digester circulation pump and starting to pump alkali through a cooking liq-
uor heater to the bottom of the digester. The condensate which contains the dissolved carbohydrates (i.e. by-products) is displaced from the top part of the digester to a condensate accumulator as the alkali pumping is continued. The amount of alkali needed for neutralization is dependent on the wood material. Any suitable alkali can be used in the displacement of the condensate and/or in the neutralization of digester contents. According to a preferred embodiment, in case essentially sulfur-free condensate is needed, the condensate is displaced with sodium hydroxide. It is also possible to obtain essentially sulfur-free condensate by using white liquor as the alkaline source, but in that case it is important to make sure that no white liquor (WL) goes to the condensate tank. If the sulfur content of the condensate is not a limiting factor, the displacement can be performed with white liquor and it is not so critical if some white liquor goes to the condensate tank. Typically, in the displacement operation, first alkaline liquid is conducted into the digester from the bottom thereof in an amount, which is needed to neutralize the digester contents and is less than the digester free volume. Thereafter the displacement is continued with hot black liquor and this stage is actually the first cooking stage. The outcoming hydrolysate is directed to the condensate accumulator and as the first displacement alkali starts to come out the outcoming liquid is conducted into the hot black liquor (HBL) accumulator. After the displacement of the condensate i.e. in the end of the recovery step the pH of the digester contents typically varies between pH 9 - 12.

After the recovery step the pH of the digester contents is increased to over 10, typically to the pH between 11 and 12, in order to continue with alkaline kraft cooking step. Thus, after the required volume of alkali (depending on the wood species) has been pumped, the process continues by pumping hot black liquor (HBL) from the HBL accumulator through cooking liquor heater. The displaced liquid is sent to the HBL accumulator while the HBL displacement continues.

The alkaline Kraft cooking is carried out with alkali rich HBL or by using a hot white liquor after the HBL stage. Typically the need for extra heating is minimal as the digester is filled with hot liquor. If heating is required, direct steam nozzles in the circulation line are used for that purpose. Cooking continues to the required H-factor.
After reaching the target H-factor the cooking is stopped by introducing cool displacement liquor to the bottom of the digester. This process step can be called as terminal displacement. The displacement liquor (DPL) is filtrate from the first brown stock washing stage. The DPL displaces the used cooking liquor from the digester to the HBL accumulator. By this way the energy and chemicals can be stored/ transferred from one cook to another. This is also an efficient way to terminate the cook.

The temperature of the digester contents is below the boiling point after the displacement is finished and the emptying of the digester is carried out with a pump. Dilution liquor is added to the digester in order to improve discharge. The dilution liquor is taken from the displacement liquor tank. The temperature is low and thus no strong gases are formed. An atmospheric discharge tank is used and the vent gases are connected to gas handling system. New cooking cycle can be started after finishing the discharge step, i.e. the digester is empty.

In order to operate the process according to the invention the tank farm area (see figure 1) typically comprises three accumulators (hot water, condensate and hot black liquor i.e. HBL accumulator) and four atmospheric tanks (displacement i.e. DPL tank, discharge tank, digester and NaOH/white liquor (WL) tank).

In the process according to figure 1, the HBL accumulator is used to storage the alkaline Kraft liquors used in hot liquor fillings and it acts as a receiving tank for used cooking liquor from the terminal displacement. Excess liquor from this tank is sent to evaporation through heat exchanger and a liquor filter.

According to the embodiment shown in Fig. 1, the incoming water is first heated with hot black liquor from HBL accumulator and then with condensate from condensate accumulator prior directing to the hot water (HW) accumulator, the heat exchangers are shown in figure 1. The fibres are separated with a liquor filter. The HBL accumulator has a circulation line where the HBL temperature is controlled with MP-stream when no hot fill is in operation.
The condensate accumulator is used as a receiving tank for the prehydrolysis stage condensate. The circulation line is used to control the pH in the accumulator. Addition of NaOH is possible if required. The condensate is cooled with the aid of a heat exchanger before sending to further processing.

The hot water accumulator is a storage tank for hot water coming from the heat exchangers. The circulation line of the hot water accumulator is equipped with a possibility to heat the water with MP-steam to the required temperature. The hot water is used in the recovery step as described above.

The displacement liquor tank is used to store washing filtrate for terminal displacement and for discharge dilution.

As shown in figure 1 there are also storage tanks for NaOH and/or white liquor.

NaOH can be used to neutralize the condensate in the circulation line of the condensate accumulator. NaOH is needed in the digester after the prehydrolysis stage. NaOH/white liquor can also be used to control the alkalinity of the HBL used in the cooking stage.
CLAIMS

1. Process for recovering by-products in a displacement batch pulping process in a digester comprising a bottom, a top and suction screens said method comprising prehydrolyzing of chips in a steam phase to produce hydrolysate and steam condensate characterized in that after the prehydrolysis stage the process continues by,
   - introducing hot washing liquid into the digester
   - while adding the washing liquid, circulating it via the suction screens to the top and to the bottom of the digester
   - continuing the circulation until the prehydrolyzed chips are under the washing liquid
   - recovering the hot washing liquid containing by-products from the digester and neutralizing the digester contents by displacing the washing liquid with alkaline liquor.

2. Process for recovering by-products according to claim 1, wherein the washing liquid is hot water.

3. Process for recovering by-products according to claim 2, wherein sodium hydroxide is added to the hot water prior introducing it into the digester.

4. Process for recovering by-products according to any of the preceding claims, wherein the hot washing liquid has a temperature of 100 - 170°C, preferably 140 - 160°C.

5. Process for recovering by-products according to any of the preceding claims, wherein the alkaline liquor is NaOH.
Fig. 1
**INTERNATIONAL SEARCH REPORT**

**International application No**

PCT/FI2011/05Q206

**A. CLASSIFICATION OF SUBJECT MATTER**

INV. D21C11/00

ADD.

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

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

D21C

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

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

EPO-Internal, WPI Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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

* Special categories of cited documents:

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**Date of the actual completion of the international search**

12 May 2011

**Date of mailing of the international search report**

04/07/2011

**Name and mailing address of the ISA/**

European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel: (+31-70) 340-2040, Fax: (+31-70) 340-3016

**Authorized officer**

Hindia, Evangelia
**INTERNATIONAL SEARCH REPORT**

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