We, MO OCH DOMSJO AKTIEBOLAG, a Swedish Company, of Box 500, 891 01 Ornskoldsvik, Sweden, hereby apply for the grant of a Patent Of Addition for the invention entitled "A METHOD FOR THE REDUCTION OF THE RESIN CONTENT DURING THE MANUFACTURE OF CELLULOSE PULPS FROM LIGNOCELLULOSIC MATERIALS" which is described in the accompanying complete specification.

We request that a Patent may be granted as a Patent Of Addition to the Patent applied for on application no. 62195/80 in the name of Mo Och Domsjo Aktiebolag.

The application is a further application made by virtue of sub-section (1) of Section 51 of the Patents Act 1952-1979 in respect of an invention disclosed in the complete specification lodged in respect of application no. 62195/80.

We request that the term of the Patent of Addition be the same as that of the patent for the main invention or so much of the term of the Patent for the main invention as is unexpired.

This application is a convention application and is based on the application numbered 80 08574-9 for a Patent or similar protection made in Sweden on the 5th December, 1980.

Our address for service is: c/- R.K. MADDERN & ASSOCIATES, 97 King William Street, Adelaide, in the State of South Australia, 5000, Australia,

Dated this 25th day of November, 1981.

MO OCH DOMSJO AKTIEBOLAG

By its Patent Attorneys,
R.K. MADDERN & ASSOCIATES

To: Commissioner of Patents,
WODEN, A.C.T. 2606
In support of the Convention application made by

MO OCH Domsjö Aktiebolag

for a patent of addition

"A METHOD FOR THE REDUCTION OF THE RESIN CONTENT DURING THE MANUFACTURE OF CELLULOSE PULPS FROM LIGNOCCELLO- LUSIC MATERIALS"

To the Commissioner of Patents,
Commonwealth of Australia.

Hans Tydén
Patent Manager

When preparing pulp according to the sulfite process the wood therefore is always stored a certain time before it is introduced into the digester and
Claim 1. A method for the reduction of the resin content in the preparation of cellulose pulp from lignocellulosic materials, whereat the lignocellulosic material is subjected to separation of the fibres, washing, possibly screening and possibly delignifying bleaching and in the form of cellulose pulp is brought to a pulp concentration of 15-35%, in one or more concentrating devices and chemicals, including alkali are added, whereafter the pulp is subjected to a mild, mechanical treatment in a device for treatment at high consistency, which device is provided with screws rotating in relation to each other under an input of energy of from 8 to 100 kWh per ton of pulp and thereafter is brought to react with the chemicals added in a separate container during a time of 0.1 to 5 hours at a substantially unchanged pulp concentration, characterised in that, the addition of chemicals consists of oxidizing bleaching agents and alkali in an amount, counted as NaOH, of 0.5 to 17 gram per kg water present.
The following statement is a full description of this invention, including the best method of performing it known to us.

"A METHOD FOR THE REDUCTION OF THE RESIN CONTENT DURING THE MANUFACTURE OF CELLULOSE PULPS FROM THE LIGNOCELLULOSIC MATERIALS"
The present invention relates to a method for the reduction of the resin content of cellulose pulps when preparing these pulps from lignocellulosic materials. The expression cellulose pulps in this description preferably is intended to mean chemical pulps, i.e. pulps prepared by any of the chemical digestion methods. Primarily the invention is applicable in the preparation of sulfite pulp but also the preparation of kraft pulp from hardwood, such as birch, is an important area for the application of the invention.

It is also possible to practice the invention in manufacturing of pulps in a higher yield than the yield of chemical pulps, as semi-chemical, thermomechanical and mechanical pulps.

State of the art

The starting material for the preparation of cellulose pulp, is lignocellulose, for instance in the form of wood, and it always contains greater or smaller amounts of resin. It is desired to remove the resin during the pulp preparation procedure to the greatest possible extent so that the fully treated pulp obtains a low resin content. High contents of resin in the finished pulp may cause problems in the use of the pulp (for instance in paper manufacture) and impair the quality of the end product. In addition the fact is that the resin complicates the very procedure of pulp preparation.
When preparing pulp according to the sulfite process the wood therefore is always stored a certain time before it is introduced into the digester and digested to cellulose pulp. During storage, so called seasoning, a change in the physical character of the resin occurs, that leads to the amount of resin in the wood being somewhat decreased and also gives rise to changes in the resin itself so that it becomes more easily dissolved during the pulp preparation procedure. The storage of the wood may be carried out in different ways. For example the wood in the form of logs may first be stored in water (floating and towing) after which the logs in the form of bundles are stored on land in a wood-yard. After a storage time of about one year the logs are taken into the pulp mill for cutting into chips and further treatment to pulp. Another method is to cut the logs to chips already when they arrive to the pulp mill and then store the chips in a pile. Treating the wood in this way one can decrease the storage time to about 3 months. Regardless of the method of stage the treatment always means a cost, and to this comes the fact that a certain loss of wood is obtained simultaneously as considerable amounts of money are tied up.

In spite of the storage the wood still contains considerable amounts of resin, though in a slightly changed form compared to the resin in the fresh wood. The larger part of the remaining resin-content is removed in different stages during the pulp preparation procedure. To remove all resin from the pulp is difficult and, above all, expensive. Finished pulp therefore almost without exception contains a certain amount of resin. During the very digestion of the wood some part of the resin is dissolved and removed when washing and screening the pulp.

The final adjustment of the resin content of the pulp is carried out in the bleaching department.
It is primarily in the alkaline stage of the bleaching sequence that resin is removed.

The final lowering and adjustment of the resin content in the pulp is usually done by means of chlorine dioxide. In sulfite mills it is common to use the bleaching sequence chlorine (C), alkali (E), hypochlorite (H), and chlorine dioxide (D), that is C-E-H-D. By varying the amount of alkali, usually sodium hydroxide, in the E-stage one can extract greater or smaller amounts of resin. Dispersing agents are often added together with sodium hydroxide in the E-stage in order to keep the resin in dispersed form (and not agglomerate) so that it can be washed out of the pulp to the utmost possible extent in the washing step following the E-stage. The final adjustment of the resin content usually is carried out in the D-stage, that is to say by varying the amount of chlorine dioxide added. The resin is separated from the pulp in the washing stage following the D-stage.

When resin problems occur in the mill (for instance foaming and clogging) it can be necessary to decrease the amount of chlorine in the C-stage, and correspondingly increase the amount of chlorine dioxide. As is well-known in the art the chlorination of the resin means that it will be more difficult to handle. The great disadvantage with the use of considerable amounts of chlorine dioxide to handle resin problems is the high price of this chemical.

When preparing pulp according to the kraft process the wood is not stored to any appreciable degree. To master resin problems in the preparation of, for instance, birch kraft pulp it is important that the logs are debarked carefully since the bark and, above all, the cambium-layer between the bark and the wood contains large amounts of resin. As in the sulfite cook a dissolution of resin takes place during the kraft cook. In order to keep the resin in dispersed form during the digestion (and to the digestion step, and this to evaporative recovery boiler).

In the possible to achieve of varying a bleaching sequence to the expense for the final.

In this thus require quality debark of the expense bleaching difficulties problems. By a finished pulp content is, market.

By a so called w the procedure decrease th some degree

The previously become the pr reduction c used regul lignocellul (free-layer screening,
digestion (so as to avoid clogging) tall oil is added to the digester. The resin extracted during the cook is separated from the pulp in the subsequent washing step, and thus goes together with the black liquor to evaporation and then to combustion in the soda recovery boiler.

In the preparation of kraft pulp it is not possible to adjust the resin content by the addition of varying amounts of alkali in an alkali stage in the bleaching sequence. Rather one is entirely restricted to the expensive bleaching chemical chlorine dioxide for the final adjustment of the resin content.

In the preparation of birch kraft pulp one is thus required to make expensive investments in high-quality debarking equipment and/or to add great amounts of the expensive chemical chlorine dioxide in the bleaching department in order to overcome the resin problems. Even if these expensive steps are taken it is difficult to reach the low resin contents of the finished pulp that are desired. Pulp of low resin content is, as is well known, much coveted on the market.

By addition of different surface active agents, so called wetting agents, at different occasions of the procedure of preparation it is also possible to decrease the resin content of the cellulose pulp to some degree.

The above stated methods were the methods which previously were the most common ones in order to overcome the problem with resin when manufacturing cellulose pulp in practice. Quite recently a method for the reduction of the resin content in cellulose pulp is being used regularly within the cellulose industry. The lignocellulosic material is subjected to separation (free-laying) of the fibres, washing, possibly screening, and possibly delignifying bleaching. The
new and characterizing for the method is, that the cellulose pulp at that moment in the pulp manufacturing process is subjected to a chemical mechanical treatment. The cellulose pulp is brought to a pulp concentration of 15 to 35% and alkali is added in an amount of 2-17 g per kg water accompanying the pulp, counted as NaOH. The cellulose pulp is then subjected to a mild, mechanical treatment in a device adapted for high consistency treatment provided with screws able to rotate relative to each other with an energy input of from 8 to 100, preferably 10 to 75 kWh per ton of pulp. Finally the cellulose pulp is brought to react with the alkali added during a time of 0.1 to 5 hours at a substantially unchanged pulp concentration in a separate container. After that treatment with the intention of decreasing the resin content of the cellulose pulp the pulp usually is transported to further treatment stages in a bleachery in order to obtain a final brightness, which usually exceeds 90% ISO. It is also possible to terminate the manufacturing of pulp with the treatment described above, which means the manufacturing of unbleached or slightly bleached cellulose pulp. The method mentioned above has shown to be very successful in the manufacturing of pulp with desired (i.e. low) content of resin in practice even in that case, that fresh wood is brought to the cooking department for cooking and separation of the fibres. Also other pulp characteristics, such as the purity of the pulp, is improved when using the new method. The method is further thoroughly described in the Swedish patent application 79 07557-8.

Description of the invention

Technical problem
When using the method according to the Swedish
patent application 79 07557-8 for manufacturing of in
the first place unbleached or slightly bleached cellu-
lose pulp it has been shown, that the brightness of the
pulp is not on a level with the high quality of the
pulp concerning the resin content.

The solution

The present invention solves this problem and
relates to a method for the reduction of the resin
content in the preparation of cellulose pulp from ligno-
cellulosic materials, whereat the lignocellulosic material
is subjected to separation of the fibers, washing,
possibly screening and possibly delignifying bleaching
and in the form of cellulose pulp is brought to a
pulp concentration of 15 to 35%, preferably 19 to 29%,
in one or more concentrating devices and chemicals,
including alkali, are added, whereafter the pulp is
subjected to a mild, mechanical treatment in a device
for treatment at high consistency, which device is
provided with screws rotating in relation to each other
under an input of energy of from 8 to 100 kWh, preferably
from 10 to 75 kWh per ton of pulp and thereafter is
brought to react with the chemicals added in a separate
container during a time of 0.1 to 5 hours at a sub-
stantially unchanged pulp concentration, which is
characterized in that the addition of chemicals consists
of oxidizing bleaching agents and alkali in an amount,
counted as NaOH, of 0.5 to 17 gram per kg water present.

The measures according to the invention are
established after that the lignocellulosic material
is digested to cellulose pulp in a digester by means
of digestion chemicals and is liberated from the spent
digestion liquor in the washing department. When the
pulp has left the washing department it usually has a
concentration of 4-6%. Usually the pulp also is screened

For comparison the partially screened pulp was
subjected to treatment with both of the chemicals, NaOH
and NaClO, in a conventional way. A certain amount of

...
subject the pulp to a mild, delignifying bleaching with some bleaching agent, for instance chlorine and/or chlorine dioxide before it is subjected to the measures of the invention.

According to the invention the starting pulp is dewatered in one or more stages so that a concentration of 15-35%, preferably 19-29%, is obtained. Usually the concentration of the pulp is carried out in one stage and suitable dewatering devices are drum washers, belt washers, roll presses and screw presses. Whether the concentration of the pulp is carried out in one or more (for example two) stages depends to some extent on whether the process of the invention is applied in an already existing mill or the process is adopted in a new or rebuilt mill. In mills already existing one usually has drum washers or thickeners installed after the screening department that raise the pulp concentration from the interval 0.5 to 3%, common in the screening department, to the interval 10 to 13%. It is, however, not necessary that the drum washer has such a dewatering capacity but a very simple drum washer which raises the pulp concentration to 4% or more will suffice. After passage over the drum washer or thickener the pulp is carried to a device in which the final dewatering to a pulp concentration of 15-35% takes place. A preferred device is a screw press. In order to facilitate dewatering of the pulp the pH-value of the incoming pulp may be adjusted to 7-9 by the addition of alkali.

Even the untreated, i.e. the partially screened pulp was tested. The results are evident from Table 8 below.
After the dewatering stage alkali and oxidizing bleaching agents are added to the pulp. The amount of alkali which are added to the pulp is of greatest importance and the added amount shall be adjusted in such a way, that the content of alkali, counted as NaOH, amounts to 0.5 to 17 gram per kg water present in the pulp suspension or, in the case that it is doubtful if a suspension exists, water accompanying the pulp. As, alkali sodium hydroxide is preferred, but it is possible to add another alkali, as potassium hydroxide, oxidized white liquor, green liquor and sodium carbonate separately or in a mixture.

Among oxidizing bleaching agents peroxide bleaching agents are preferred and especially hydrogen peroxide is preferred. But even hypochlorite bleaching agents as sodium hypochlorite can be used to advantage. Even other oxidizing bleaching agents can be considered. The addition of oxidizing bleaching agents is adjusted so that the amount of bleaching agents is 0.2 to 22, preferably 0.3 to 11 gram per kg water present.

In some cases it is advantageous to add other chemicals to the pulp, such as surface active agents (so called wetting agents) and complex-forming substances besides alkali and oxidizing bleaching agents.

After this the pulp is subjected to a mild mechanical treatment in a device suited for high-consistency treatment which device is provided with screws rotating in relation to each other under such conditions that the input of energy will be from 8 to 100 kWh per ton of pulp, and preferably from 10 to 75 kWh per ton of pulp. A suitable apparatus for such treatment is a screw defibrator (screw refiner) and especially suitable is the screw defibrator sold by MoDoMekan AB under the Trade name FROTAPULPER®. This screw defibrator consists principally of two...
rotary screws which are arranged parallel to each other in a housing provided with an inlet and an outlet. The screws engage into each other for the purpose of kneading of the feed material and the screw flights are provided with indentations on the circumference of at least some flights of the screws in order to form teeth between the indentations. Such a screw defibrator is described in the U.S. patent specification 3 064 908. The pulp mixed with chemicals is subjected to shearing and kneading forces in the screw defibrator in form of pulsating pressure loads. As a result of this treatment a very effective impregnation of the pulp with the added chemicals is obtained. As for the pulp fibres the treatment is mild, since the fibres are not shortened (which is the case in beating or milling) or in any other way negatively influenced. The treatment in the screw defibrator usually is carried out at atmospheric pressure but it can also be carried out at superatmospheric pressures of up to 500 kPa. During the mechanical treatment the temperature of the pulp is raised since at least 60% of the energy put in is transformed to heat. The higher the input of energy, the greater is the temperature increase during the work.

After the mild mechanical treatment the pulp is transferred by means of a suitable device, such as a pump, screw feeder or belt conveyor to a tower or similar container for continued reaction with the added chemicals (mainly alkali) at desired temperature. The retention time for the pulp in this stage can vary between 6 minutes and 5 hours.

After this the pulp is washed on known washing apparatus so that the resin extracted from the pulp is removed from the pulp.

According to a preferred embodiment of the invention one applies a short retention time between the
dewatering stage and the mild mechanical treatment. The short retention time suitably is established by transporting the pulp through a screw feeder. The retention time should lie within the interval of from 2 to 10 seconds. Besides using a screw feeder it is also possible to let the pulp pass through a so called mixer for chemicals, which is an apparatus used for mixing chemicals into pulp.

In the treatment of certain pulps it is necessary to raise the reaction temperature of the mild mechanical treatment of the pulp and the temperature of the subsequent reaction with the chemicals added in the retention tower in excess of the temperature elevation which is caused by the kneading and shearing work in order to reach the resin removal intended. In such cases steam is added to the pulp and the addition of steam should be carried out during the short retention time.

The addition of chemicals and steam to the pulp lowers the pulp concentration. The concentration of pulp must not, however, be lower than 15% when the pulp is subjected to the mild mechanical treatment.

After that the pulp is treated according to the invention and has been subjected to washing in order to remove the dissolved resin from the pulp the pulp is directly transported to drying or to final manufacturing, for example to paper of different qualities. The method according to the invention is consequently applicable in the first place to unbleached or slightly bleached cellulose pulp. However, it is fully possible to apply the method according to the invention also to pulp, which is bleached to a varying extent, including bleaching to a brightness of the pulp exceeding 90% ISO.

Advantages

By subjecting the cellulose pulp to the treatment

The data of analysis above show, that the method according to the invention, gives a pulp with a higher brightness especially in extract content compared to the
according to the invention and regulating the amount of chemicals added, the temperature and the input of energy it is possible to adjust the resin content as well as the brightness of the pulp to the desired levels in an effective way. Regarding the adjustment of the resin content of the pulp it is in the first place increased temperature and increased input of energy, which lead to increased dissolving of resin from the pulp. An increase in the amount of alkali added does not have as great an influence on the resin dissolving as the above mentioned parameters. At a certain pulp there is an optimal amount of addition of alkali. Additions exceeding this amount has no or little influence on the dissolving of the resin. Such an addition of alkali in excess can have a negative influence on other pulp qualities. Regarding the brightness of the pulp it is in the first place the amount of addition of an oxidizing bleaching agent, which determines the level of brightness which is obtained.

The present invention can be utilized in different ways. In a sulfite mill it has been found that the present invention makes it possible to leave out the storage of wood and bring fresh wood directly into the mill. As a result, even if the cost of the equipment needed for the process of the invention is included, the costs for the preparation of sulfite pulp will be considerably reduced. Even if the storage of wood at a sulfite mill is retained the process of the invention is of great value since one is able to regulate the resin content of the finished pulp in a totally different and better way than has been possible before.

In a Kraft mill it will be possible through the
invention, for example, to prepare birch kraft pulp of an even and low resin content which previously has not always been possible. Furthermore, one can in the preparation of such pulp lower the requirements for debarking of the birch wood and decrease the addition of the expensive chemical chlorine dioxide which also is advantageous from an environmental pollution point of view.

The advantages of the process of the invention are also shown in the examples following later in the description.

Description of drawings

Figure 1 shows a suitable apparatus for a preferred embodiment of the process of the invention.

Best mode

A plurality of tests according to the invention and comparing tests have been made. How these tests have been performed and the results which have been obtained is shown in the following examples.

Example 1

A screened spruce sulfite pulp of paper pulp grade was treated according to the invention by using the apparatus shown in Figure 1.

The characteristics of the screened spruce sulfite pulp measured according to SCAN-tests are shown in Table 1.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>SCAN-C 1:59</th>
<th>SCAN-C 2:61</th>
<th>SCAN-C 15:62</th>
<th>SCAN-C 7:62</th>
<th>SCAN-C 11:62</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kappa number</td>
<td>12.2</td>
<td>78.2</td>
<td>1.93</td>
<td>69.2</td>
<td></td>
</tr>
<tr>
<td>Viscosity, dm³/kg</td>
<td></td>
<td></td>
<td>1073</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Extract content DCM, %</td>
<td>SCAN-C 1:59</td>
<td>SCAN-C 2:61</td>
<td></td>
<td>SCAN-C 7:62</td>
<td>SCAN-C 11:62</td>
</tr>
</tbody>
</table>

The claims defining the invention are as follows:

1. A method for the reduction of the resin content
The screened pulp with a temperature of 62°C was passed through conduit 1 to a screw press 2, in which the pulp was dewatered to a concentration of 29.5%. The water that had been pressed out was removed through the conduit 3. At the outlet of the screw press 2 alkali in the form of sodium hydroxide (NaOH) was added to the pulp in an amount of 1.0%, counted on absolutely dry pulp. This addition resulted in an alkali amount of 4.2 g NaOH per kg water present. The alkali was stored as a water solution in the container 4 and was passed to the screw press 2 through the conduits 5 and 6. From the screw press 2 the pulp was passed through the conduit 7 to a screw feeder 8. By means of the screw feeder 8 the pulp was passed to a screw defibrator 9, which was of the type that is sold by MoDoMekan AB under the Trade name FROTAPULPER™. Just before the screw defibrator 9 hydrogen peroxide $\text{H}_2\text{O}_2$ was added to the pulp in an amount of 0.21%, counted on absolutely dry pulp. This addition resulted in an amount of peroxide of 0.9 g $\text{H}_2\text{O}_2$ per kg water present. The hydrogen peroxide in the form of a water solution was stored in the container 10 and was passed to the pulp through the conduits 11 and 12. In the screw defibrator 9 the pulp was subjected to kneading and shearing work corresponding to an energy input of 28 kWh per ton of pulp. As a result the temperature of the pulp rose to 69°C. After this the pulp was allowed to fall by gravity through a vertical shaft and a conduit 13 to a tower 14. In the tower 14 the reactions between the pulp and the chemicals NaOH and $\text{H}_2\text{O}_2$ was carried to a completion. After 120 minutes a sample of the pulp was taken, whereafter the sample was washed, dried and analysed. The results of the analysis are clear from Table 3.
The above described test was repeated with the only difference, that no hydrogen peroxide was added to the pulp (0-test). Also this pulp was washed, dried and analysed. The results of the analysis are clear from Table 2.

Table 2

<table>
<thead>
<tr>
<th>Pulp characteristics</th>
<th>0-test</th>
<th>According to the invention</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kappa number</td>
<td>9.7</td>
<td>8.0</td>
</tr>
<tr>
<td>R18, %</td>
<td>78.3</td>
<td>78.3</td>
</tr>
<tr>
<td>Viscosity, dm$^3$/kg</td>
<td>1070</td>
<td>1066</td>
</tr>
<tr>
<td>Extract content DCM, %</td>
<td>0.27</td>
<td>0.24</td>
</tr>
<tr>
<td>Brightness ISO, %</td>
<td>66.0</td>
<td>75.9</td>
</tr>
</tbody>
</table>

As is evident from the Table above a higher brightness was obtained at the method according to the invention compared to the 0-test. Furthermore the Kappa number of the pulp was decreased more according to the invention compared to the 0-test while maintaining the same viscosity. The resin content is very low for both of the pulps.

Example 2

At the same time as the tests above were made on a large scale samples of the screened spruce sulfite pulp of paper pulp grade were taken for tests in the laboratory. In these tests the chemicals NaOH and NaOH + H$_2$O$_2$ respectively were added to the pulp in a conventional way. A certain amount of pulp was in test 1 passed to a treating vessel, which was kept in a water bath at 69°C. 1% NaOH counted on absolutely dry pulp was in the form of a water solution mixed into the pulp by means of a propeller stirrer. The pulp concentration was 12%. This addition meant, that the amount of NaOH was 1.4 gram per kg water present. During 120 minutes the pulp was permitted to react with the sodium hydroxide, whereafter the pulp was washed, dried and analysed.
In test 2 the same thing was done, with the only difference, that also hydrogen peroxide $\text{H}_2\text{O}_2$ was added together with NaOH. Hydrogen peroxide in the form of a water solution was added in an amount of 0.21%, counted on absolutely dry pulp. This addition meant that the amount of $\text{H}_2\text{O}_2$ was 0.3 g per kg water present. The test was finished off by washing, drying and analysing the pulp. The results of the analysis from the tests 1 and 2 and from the test according to the invention in Example 1 are evident from Table 3.

Table 3

<table>
<thead>
<tr>
<th>Pulp characteristics</th>
<th>Test 1</th>
<th>Test 2</th>
<th>According to the invention</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaOH</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Kappa number</td>
<td>10.4</td>
<td>9.7</td>
<td>8.0</td>
</tr>
<tr>
<td>R18, %</td>
<td>78.2</td>
<td>78.3</td>
<td>78.3</td>
</tr>
<tr>
<td>Viscosity, dm$^3$/kg</td>
<td>1066</td>
<td>1069</td>
<td>1066</td>
</tr>
<tr>
<td>Extract content DCM, %</td>
<td>0.69</td>
<td>0.68</td>
<td>0.24</td>
</tr>
<tr>
<td>Brightness ISO, %</td>
<td>65.7</td>
<td>72.1</td>
<td>73.9</td>
</tr>
</tbody>
</table>

As is evident from the Table the pulp manufactured according to the invention is far superior to the pulps according to tests 1 and 2 regarding the Kappa number, the extract content and the brightness. Especially big is the difference in extract content. In test 2 both of the chemicals, i.e. NaOH + $\text{H}_2\text{O}_2$, have been added to the pulp in the same amount, as in the method according to the invention. In spite of this the method according to the invention leads to a better pulp, not only as far as the resin content is concerned but also as regards the Kappa number and the brightness as commented on earlier.

These tests show, that it is not the added chemicals themselves, which explain the good qualities of the pulp, that have been treated according to the invention.
Example 3

A screened birch sulfate pulp was used for tests according to the invention and comparison test (0-test). The pulp characteristics are evident from Table 4 below.

Table 4

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kappa number</td>
<td>18.7</td>
</tr>
<tr>
<td>Viscosity, dm$^3$/kg</td>
<td>1182</td>
</tr>
<tr>
<td>Extract content DCM, %</td>
<td>0.88</td>
</tr>
<tr>
<td>Brightness ISO, %</td>
<td>31.7</td>
</tr>
</tbody>
</table>

Before the treatment according to the invention the screened pulp was subjected to a delignifying bleaching by means of chlorine and chlorine dioxide. Chlorine and chlorine dioxide were added to the pulp at the same time in amounts corresponding to 3.4% and 0.3% respectively, counted as active chlorine on absolutely dry pulp. The temperature was 40°C and the time 30 minutes. Thereafter the pulp was washed.

The partially delignified pulp in the above described way was subjected to a treatment according to the invention by using the apparatus shown in Figure 1.

The pulp having a temperature of 58°C was passed through the conduit 1 to the screw press 2, in which the pulp was dewatered to a concentration of 27.8%. The water that had been pressed out was removed through the conduit 3. At the outlet of the screw press 2 alkali in the form of NaOH was added to the pulp in an amount of 1.85%, counted on absolutely dry pulp. This addition resulted in an alkali amount of 7.1 g NaOH per kg water present. The alkali was stored as a water solution in the container 4 and was passed to the screw press 2 through the conduits 5 and 6. From the screw press 2 the pulp was passed through the conduit 7 to the screw
feeder 8. By means of the screw feeder 8 the pulp was passed to the screw defibrator 9, which was of the type that is sold by MoDoMekan AB under the Trade name FROTAPULPER®. Just before the screw defibrator 9 hydrogen peroxide H₂O₂ was added to the pulp in an amount of 0.33%, counted on absolutely dry pulp. This addition resulted in an amount of peroxide of 1.3 g H₂O₂ per kg water present. The hydrogen peroxide in the form of a water solution was stored in the container 10 and was passed to the pulp through the conduits 11 and 12. In the screw defibrator 9 the pulp was subjected to kneading and shearing work corresponding to an input of energy of 38 kWh per ton of pulp. As a result the temperature of the pulp rose to 67°C. After this the pulp was allowed to fall by gravity through a vertical shaft and the conduit 13 to the tower 14. In the tower 14 the reactions between the pulp and the chemicals NaOH and H₂O₂ were carried to a completion. After 10, 30 and 120 minutes retention time in the tower 14 samples of the pulp was taken. The samples were washed, dried and analysed. The results of the analysis are evident from Table 5.

For comparison the partially delignified pulp was subjected to treatment with both the chemicals, NaOH and H₂O₂, in a conventional way. A certain amount of pulp was passed into a treating vessel, which was kept in a water bath at 67°C. 1.85% NaOH and 0.33% H₂O₂, counted on absolutely dry pulp, were by means of a propeller stirrer mixed into the pulp. The pulp concentration was at that moment 12%. These additions of chemicals corresponded to an amount of 2.5 g NaOH per kg water present and 0.5 g H₂O₂ per kg water present. The pulp and the chemicals were then allowed to react with each other for 120 minutes, whereafter samples of the pulp were washed, dried and analysed. The results of the analysis are evident from Table 5.
Table 5

<table>
<thead>
<tr>
<th>Pulp characteristics</th>
<th>0-test</th>
<th>According to the invention, with a reaction time in the last stage of 10 min</th>
<th>30 min</th>
<th>120 min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kappa number</td>
<td>3.8</td>
<td>3.1</td>
<td>2.9</td>
<td>2.9</td>
</tr>
<tr>
<td>Viscosity, dm³/kg</td>
<td>1107</td>
<td>1110</td>
<td>1103</td>
<td>1106</td>
</tr>
<tr>
<td>Extract content DCM, %</td>
<td>0.68</td>
<td>0.26</td>
<td>0.24</td>
<td>0.23</td>
</tr>
<tr>
<td>Brightness ISO, %</td>
<td>57.2</td>
<td>58.0</td>
<td>58.8</td>
<td>60.1</td>
</tr>
</tbody>
</table>

The above stated data of analysis show, that already at short retention times after the mild mechanical treatment the method according to the invention has resulted in a lower Kappa number, higher brightness and a considerably lower content of resin compared to the conventional method.

It is also clear, that an increased retention time in the concluding stage according to the invention is favourable especially for the brightness of the pulp.

Example:

A partially screened sulfate pulp manufactured of mixed softwood containing mainly eucalyptus saligna and eucalyptus grandis was used for a test according to the invention and a comparison test (0-test). The data of analysis of the pulp are evident from Table 6 below.

Table 6

| Kappa number         | 22.2   |
| Viscosity, dm³/kg    | 1170   |
| Extract content DCM, % | 0.91   |
| Brightness ISO, %    | 34.9   |

The partial screening meant, that knots and bigger nondigested pieces of wood were separated from the pulp, but that material which normally is classified as shives was not separated.
This pulp was subjected to treatment according to the invention by using the apparatus shown in Figure 1.

The pulp with a temperature of 55°C was passed through the conduit 1 to the screw press 2, in which the pulp was dewatered to a concentration of 31.0%. The water that had been pressed out was removed through the conduit 3. At the outlet of the screw press 2 alkali in the form of NaOH was added to the pulp in an amount of 0.95%, counted on absolutely dry pulp. This addition resulted in an alkali amount of 4.3 g NaOH per kg water present. The sodium hydroxide was stored as a water solution in the container 4 and was passed to the screw press 2 through the conduits 5 and 6. From the screw press 2 the pulp was passed through the conduit 7 to the screw feeder 8. By means of the screw feeder 8 the pulp was passed to the screw defibrator 9, which was of the type that is sold by MoDoMekan AB under the Trade name FROTAPULPEAr. Just before the screw defibrator 9 sodium hypochlorite was added to the pulp in an amount of 0.55%, counted as active chlorine on absolutely dry pulp. This addition resulted in an amount of hypochlorite of 2.5 g NaCIO per kg water present. Sodium hypochlorite in the form of a water solution was stored in the container 10 and was passed to the pulp through the conduits 11 and 12. In the screw defibrator 9 the pulp was subjected to kneading and shearing work corresponding to an input of energy of 26 kWh per ton of pulp. As a result the temperature of the pulp rose to 63°C. After this the pulp was allowed to fall by gravity through a vertical shaft and the conduit 13 to the tower 14. In the tower 14 the reactions between the pulp and the chemicals NaOH and NaCIO were carried to completion. After a retention time of 120 minutes samples of the pulp were taken, which were washed, dried and analysed. The results of the analysis are evident from Table 7.
For comparison the partially screened pulp was subjected to treatment with both of the chemicals, NaOH and NaClO, in a conventional way. A certain amount of pulp was passed into a treating vessel, which was kept in a water bath at 63°C. 0.95% NaOH, counted on absolutely dry pulp, and 0.55% NaClO, counted as active chlorine on absolutely dry pulp, were by means of a propeller stirrer mixed into the pulp. The pulp concentration was at that moment 12%. These additions of chemicals correspond to an amount of 1.3 g NaOH per kg water present and 0.8 g NaClO per kg water present. The pulp and the chemicals were then allowed to react with each other for 120 minutes, whereafter a sample of the pulp was washed, dried and analysed. The results of analysis are evident from Table 7.

Table 7

<table>
<thead>
<tr>
<th>Pulp characteristics</th>
<th>0-test According to the invention</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kappa number</td>
<td>14.6</td>
</tr>
<tr>
<td>Viscosity, dm³/kg</td>
<td>1091</td>
</tr>
<tr>
<td>Extract content DCM, %</td>
<td>0.57</td>
</tr>
<tr>
<td>Brightness ISO, %</td>
<td>43.9</td>
</tr>
</tbody>
</table>

The above stated data of analysis show, that the method according to the invention gives a pulp with a lower Kappa number, higher brightness and considerably lower resin content compared to pulp treated according to a conventional method even when the oxidative bleaching agent consists of sodium hypochlorite.

The two pulps obtained in the described way were also tested in order to detect their content of impurities. Samples of the pulps were screened on a screen of the type Sommerville and the amount of material which was retained on a screen plate with a slot size of 0.15 mm was measured.
Even the untreated, i.e. the partially screened pulp was tested. The results are evident from Table 8 below.

Table 8

<table>
<thead>
<tr>
<th>Original pulp</th>
<th>0-test</th>
<th>According to the invention</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content of shives, % on absolutely dry pulp</td>
<td>0.72</td>
<td>0.61</td>
</tr>
</tbody>
</table>

From the values stated above are evident, that the method according to the invention is particularly effective also as far as elimination of particles from the pulp is concerned.

In the above described Examples alkali in the form of sodium hydroxide has always been added to the pulp at the outlet of the screw press 2 via the conduits 5 and 6 in Figure 1. The oxidative bleaching agent has always been added just before the screw defibrator via the conduits 11 and 12.

However, it is possible and advantageous to add alkali as well as an oxidative bleaching agent in a lot of places in the method according to the invention. For example alkali can be added to the pulp in the screw feeder 8 via the conduit 5. Furthermore alkali can be added to the pulp in the screw defibrator 9 via the conduit 15. It is also possible, to divide the addition of alkali into many, possibly all the above stated places of addition. The oxidative bleaching agent can be added to the pulp in the screw defibrator 9 via the main conduit 11. It is also possible to add the oxidative bleaching agent to the pulp in the screw press 2 via the conduits 11 and 16 and in the screw feeder 8 via the conduits 11, 16 and 17. Corresponding to the addition of alkali the oxidative bleaching agent can be divided into many, possibly all the above mentioned places of addition.
If necessary to increase the temperature considerably steam can be added to the pulp in the screw feeder 8 via the conduit 18.

Example 5

5. A screened stone groundwood pulp manufactured of wood of spruce was used for a test according to the invention as well as for a comparison test (0-test). In order to remove heavy metals the pulp was treated with 0.2% (counted on absolutely dry pulp) diethylenediamine-pentaacetic acid at a temperature of 65°C for a time of 2 hours. The pulp characteristics are evident from Table 9.

At the test according to the invention the apparatus shown in Fig. 1 was used.

The pulp with a temperature of 50°C was passed through the conduit 1 to the screw press 2, in which the pulp was dewatered to a concentration of 31%. The water that had been pressed out was removed through the conduit 3. At the outlet of the screw press 2 a mixture of alkali and sodium silicate in an amount of 1.8% NaOH and 4.0% Na₂SiO₃, counted on absolutely dry pulp, was added to the pulp. This addition corresponds to 8.1 g NaOH and 18 g Na₂SiO₃ per kg water present. The chemical mixture of sodium hydroxide and sodium silicate was stored as a water solution in the container 4 and was passed to the screw press 2 through the conduits 5 and 6. From the screw press 2 the pulp was passed through the conduit 7 to the screw feeder 8. By means of the screw feeder 8 the pulp was passed to the screw defibrator 9, which was of the type that is sold by MoDoMekan AB under the trade name FROTAPULPER. Just before the screw defibrator 9 hydrogen peroxide in an amount of 3%, counted on absolutely dry pulp, was added to the pulp. This addition resulted in an amount of hydrogen peroxide of 13.5 g H₂O₂ per kg water present. The hydrogen peroxide in the form of a water solution was stored in the container 10 and was added to the pulp through the conduits 11 and 12. In the screw defibrator 9 the pulp was subjected to kneading and shearing work corresponding to an input of energy of 35 kWh per ton of pulp. As a result the
Temperature of the pulp rose to 58°C. After this the pulp was allowed to fall by gravity through a vertical shaft and the conduit 13 to the tower 14. In the tower 14 the reactions between the pulp and the chemicals NaOH, Na$_2$SiO$_3$ and H$_2$O$_2$ were carried to completion. After a retention time of 120 minutes samples of the pulp were taken, which were washed, dried and analysed. The results of the analysis are evident from Table 9.

For comparison the screened and pretreated pulp was subjected to treatment with the three chemicals NaOH, Na$_2$SiO$_3$ and H$_2$O$_2$ in a conventional way. A certain amount of pulp was passed into a treating vessel, which was kept in a water bath at a temperature of 60°C. 1.8% NaOH, 4.0% Na$_2$SiO$_3$ and 3% H$_2$O$_2$, counted on absolutely dry pulp, were by means of a propeller stirrer mixed into the pulp. The pulp concentration was at that moment 12%. These additions of chemicals correspond to an amount of 2.5 g NaOH, 5.5 g Na$_2$SiO$_3$ and 4.7 g H$_2$O$_2$ per kg water present. The pulp and the chemicals were then allowed to react with each other for 120 minutes, whereafter samples of the pulp were washed, dried and analysed for brightness and extract content. The results of the analysis are evident from Table 9.

Besides brightness and extract content the different pulps (including the original pulp) were tested in order to detect their content of impurities. Samples of the pulps were screened on a screen of the type Sommerville and the amount of material which was retained on a screen plate with a slot size of 0.15 mm was measured. The results are evident from Table 9 below.

Table 9

<table>
<thead>
<tr>
<th>Pulp characteristics</th>
<th>Original pulp</th>
<th>0-test</th>
<th>Pulp acc. to the invention</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brightness ISO, %</td>
<td>65.8</td>
<td>76.9</td>
<td>79.5</td>
</tr>
<tr>
<td>Extract content DKM, %</td>
<td>1.18</td>
<td>0.71</td>
<td>0.25</td>
</tr>
<tr>
<td>Content of shives, %</td>
<td>0.16</td>
<td>0.11</td>
<td>0.06</td>
</tr>
</tbody>
</table>
The data of analysis above show, that the method according to the invention, gives a pulp with a higher brightness and essentially lower extract content compared to the pulp treated in a conventional way. It is also evident, that the method according to the invention is very effective also as far as elimination of particles is concerned.
The claims defining the invention are as follows:

1. A method for the reduction of the resin content in the preparation of cellulose pulp from lignocellulosic materials, whereat the lignocellulosic material is subjected to separation of the fibres, washing, possibly screening and possibly delignifying bleaching and in the form of cellulose pulp is brought to a pulp concentration of 15-35%, in one or more concentrating devices and chemicals, including alkali are added, whereafter the pulp is subjected to a mild, mechanical treatment in a device for treatment at high consistency, which device is provided with screws rotating in relation to each other under an input of energy of from 8 to 100 kWh per ton of pulp and thereafter is brought to react with the chemicals added in a separate container during a time of 0.1 to 5 hours at a substantially unchanged pulp concentration, characterised in that, the addition of chemicals consists of oxidizing bleaching agents and alkali in an amount, counted as NaOH, of 0.5 to 17 gram per kg water present.

2. A method according to claim 1 characterised in that the oxidizing bleaching agent is added in an amount of 0.2-22 gram per kg water present.

3. A method according to claim 2 wherein the amount of oxidizing bleaching agent added is in an amount of 0.3-11 gram per kg water present.

4. A method according to any one of claims 1 - 3, characterised in that the oxidizing bleaching agent consists of peroxide.

5. A method according to any one of claims 1 - 3, characterised in that the oxidizing bleaching agent consists of hypochlorite.
6. A method according to any one of claims 1 - 5, characterised in that the alkali consists of sodium hydroxide.

7. A method for the reduction of the resin contents in the preparation of cellulose pulp from lignocellulosic materials, substantially as hereinbefore described with reference to any one of the Examples herein.

Dated this 25th day of November, 1981.

MO OCH DOMSJO AKTIEBOLAG
By its Patent Attorneys,
R.K. MADDERN & ASSOCIATES
with the sodium hydroxide, whereafter the pulp was washed, dried and analysed.