(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number : **0 442 806 A1**

(12) EUROPEAN PATENT APPLICATION

(21) Application number : **91400355.3**

(22) Date of filing : **12.02.91**

(51) Int. Cl.^{5} : **D21C 1/04, D21C 3/26**

(30) Priority : **12.02.90 ZA 901031**

(43) Date of publication of application :
21.08.91 Bulletin 91/34

(84) Designated Contracting States :
AT BE CH DE DK ES FR GB GR IT LI LU NL SE

(71) Applicant : **SAPPI LIMITED**
48 Ameshoff Street, Braamfontein
Johannesburg, Transvaal (ZA)

(72) Inventor : **Raubenheimer, Stephanus Jacobus**
16 Pienaar Street, Dal Fouche
Springs, Transvaal (ZA)

(74) Representative : **Phélip, Bruno et al**
c/o Cabinet Harlé & Phélip 21, rue de La Rochefoucauld
F-75009 Paris (FR)

(54) A process for producing pulp from a lignocellulosic-containing material.

(57) This invention provides a process for producing pulp from a lignocellulosic-containing material.

According to one aspect of the invention, there is provided a process for reducing the lignin content (as expressed by the Kappa number) of unbleached pulp intended for dissolving grade pulp manufacture, the process including the step of applying an alkali extraction step to the pulp.

According to another aspect of the present invention, there is provided a process for producing dissolving grade pulp, the process including the following steps :

a pre-hydrolysing step ;
a step of dissolving the lignin contained in the pre-hydrolysed material ; and
carrying out an alkali extraction step.

The alkali extraction step may include using about 2% to 5%, preferably about 3%, alkali by mass of pulp ; at a temperature of about 50°C to 100°C, preferably about 70°C to 80°C ; for a period sufficient to give optimal lignin removal. The alkali used is preferably sodium hydroxide.

According to a further aspect of the present invention, there is provided a dissolving grade pulp when produced by a process described above.

Jouve, 18, rue Saint-Denis, 75001 PARIS

A PROCESS FOR PRODUCING PULP FROM A LIGNOCELLULOSIC-CONTAINING MATERIAL

This invention relates to a process for producing pulp from a lignocellulosic-containing material. More particularly, this invention relates to a process for producing dissolving pulp from a lignocellulosic-containing material.

Wood pulp containing high levels of alpha cellulose, which is used in the manufacture of various cellulose derivatives for various end uses or products, is known in the art as dissolving pulp. Other terms synonymous with dissolving pulp are chemical cellulose and special high alpha pulp. Two processes are in general use for the manufacture of dissolving pulp viz :

1) The acid sulphite process, the development of which started at about the beginning of this century (Rydholm, S.A., Pulping Processes, p. 280, Interscience Publishers, New York-London-Sydney 1965) ; and

2) The prehydrolysis - Kraft process, the development of which started in 1929 (Rydholm referred to above - p 281)

The latter process makes use of an acidic pretreatment ("prehydrolysis") step in order to remove hemicellulose prior to the alkaline pulping (delignification) step. South African Patent 88/4037 discloses the prehydrolysis - neutral sulphite - anthraquinone process (PH-NS-AO) for the manufacture of "hemicellulose hydrolysate and special pulp" (high alpha grade). This prehydrolysis step performs essentially the same function as the prehydrolysis step prior to the Kraft (Sulphate) pulping method for the manufacture of dissolving grade pulp ; while the neutral sulphite-anthraquinone delignification step is essentially the same as the process also known as the semi-alkaline sulphite-anthraquinone (SAS-AQ) process, first reported at a technical conference in 1979 by Raubenheimer, S and Eggers, S.H., (both being research workers employed by SAPPI LIMITED, the present applicant herein) viz the 11th European ESPRA Meeting, Maastricht, The Netherlands, during May 1979.

In the aforementioned patent specification, the washing of the cooked chip material is reported to have taken place over a period of 10 hours, which is clearly impractical ; and certain of the tests show a Kappa number which is not as low as could reasonably be required or expected.

It has been found by the inventor of the present invention that the aforementioned process could be improved when applied to timber presently used for dissolving pulp manufacture in South Africa viz Gum (various Eucalyptus species - mostly E. grandis) and Wattle (Acacia mearnsii) and it is therefore an object of the present invention to provide a process which overcomes, at least partly, the disadvantage(s) of the aforementioned process and hence of the prior art.

According to one aspect of the present invention, there is provided a process for producing dissolving grade pulp, the process including -

applying a prehydrolysing step to a lignocellulosic-containing material for example hardwood.
partly dissolving the lignin contained in the prehydrolysed material (- a pulping step) ; and
carrying out an alkali extraction step.

About 2% to 5%, preferably about 3%, by mass of alkali may be used to carry out the alkali extraction step.

The alkali used may preferably be sodium hydroxide.

The alkali extraction step may be carried out at a temperature of about 50°C to 100°C, preferably about 70°C to 80°C, for a period sufficient to give optimal lignin removal.

According to another aspect of the present invention, there is provided a process for reducing the lignin content (as expressed by the Kappa number) of unbleached pulp intended for dissolving grade pulp manufacture, the process including applying an alkali extraction step to the pulp.

About 2% to 5%, preferably 3%, by mass of alkali may be used to carry out the alkali extraction step.

The alkali used may preferably be sodium hydroxide.

The alkali extraction step may be carried out at a temperature of about 50°C to 100°C, preferably 70°C to 80°C, for a period sufficient to give optimal lignin removal.

Spent liquor from the pulping step may be removed from a digester, treated with alkali, and re-introduced to the digester to lower the Kappa number of the pulp and/or to improve evaporation of the spent liquor.

After re-introduction of spent liquor to the digester, the digester may be operated at a temperature > 100°C and at elevated pressure.

About 10% to 15% alkali by mass of pulp may be used.

The alkali used may preferably be sodium hydroxide.

The invention extends to a dissolving grade pulp whenever produced by a process as herein described and/or exemplified.

The invention will now be described and illustrated in greater detail with reference to the following nonlimiting examples :

EXAMPLE 1

Prehydrolysis and pulping were carried out under laboratory conditions on run-of-mill (ENSTRA) Eucalyptus wood chips. The prehydrolysis conditions and yields are shown in Table 1 hereunder.

Table 1

Run No.	Hydrolysis agent % on wood	Time to temp. (mins)	Time at temp. (hrs)	Yield (%)
P1	2% - H_2SO_4	90 to 155 °C	2 at 155 °C	73,1
P2	1% - SO_2	60 to 165 °C	2 at 165 °C	70,5
P3	0,25% - SO_2	60 to 165 °C	2 at 165 °C	80,4
P4	0,25% - SO_2	60 to 165 °C	3 at 165 °C	77,6
P5	0,2% - SO_2	60 to 150 °C	2 at 150 °C	85,3

(P1 - P4 used 4,0/1 L/W ratio; P5 used 5,0/1 L/W ratio)

Thereafter pulping was carried out on material from three of the prehydrolysis runs, yielding the pulping results shown in Table 2 hereunder.

Table 2

Cook No.	Prehydrolysis Conditions	Charge as Na_2O(%)			Yield (%)		Kappa No.
		Na_2SO_3	Na_2CO_3	NaOH	Screened	Rejects	
C1	AsP3 - Table 1	17	4	-	51,97	0,1	42,5
C2	AsP4 - Table 1	20	2,5	2,5	49,44	0,02	87,9
C3	AsP5 - Table 1	17	4	-	50,96	0,52	32,9

The following process conditions applied :

0,2% Anthraquinone in all cases
3,5/l L/W ratio in all cases
85 mins to temperature (175°)in all cases
3 hours at 175°C for C1 and C3
5 hours at 175°C for C2

The Kappa number from cook no. C1 was relatively high.

Virkola has reported Kappa numbers in the low twenties after the pulping step but how such figures were obtained is uncertain. [Virkola, N-E and Tikka, P.O. - Tappi Pulping Conference, 1988 ; New Orleans U.S.A. October 30 - November 3 p 255 . In South Africa patent No. 88/4037 (with Virkola and Tikka as inventors) continuous washing for 10 hours after pulping is reported].

For cook number C2 the chemical charge was increased, caustic soda was added and the pulping time was increased significantly in order to reduce the Kappa number which, contrary to expectations, increased. It is believed that this increase was due to the harsher prehydrolysis conditions resulting in higher deactivation of the lignin.

Cook number C3 gave a lower Kappa number than C1 possibly because of the milder prehydrolysis conditions.

In order to reduce the Kappa number a caustic extraction step was applied.

This yielded the results shown in Table 3 below.

Table 3

Cook No.	Extraction No.	Extraction medium	Kappa No.		Yield on unbl. pulp (%)
			Before	After	
C2	E1	3% NaOH*	87,9	26,6	N.D.
C2	E2	Water	87,9	66,6	N.D.
C1	E3	3% NaOH	42,5	16,9	N.D.
C3	E4	3% NaOH	32,9	.17,6	95,71

The following process conditions applied:

* - Percentage on pulp

Time - 1 hour

Temperature - 70°C

Consistency - 10%

It was apparent that some of the residual lignin in the unbleached pulp was rather loosely held and could be removed to a large extent with a caustic soda extraction step (followed by washing).

Two bleaching runs were done, the results of which are shown in the following table :

Table 4

Unbl. pulp	Bl. No.	Sequence	Brightness (%)	Alpha Cellulose %	Yield %		Visc. cm^3/g
					Bl.	On Timber	
C1	B1	C3,5 - E4,5 - D1,5	87,6	95,50	89,37	37,3	528
E4	B2	C3,5 - E2,2 - D0,8	89,5	96,56	97,36	40,5	846

Bleach number B1 was done before the positive effect of an extraction step after pulping was established.

Because of the high unbleached Kappa number, bleach chemical charges were extremely high. Both the alpha-cellulose content and the pulp yield looked promising. Bleach number B2 was done using an extracted unbleached pulp with a kappa number of 17,6. Both the alpha cellulose content and pulp yield were high.

EXAMPLE 2

RESULTS OBTAINED WITH EUCALYPTUS AND WATTLE WOOD (RUN-OF-MILL - SAICCOR)

PREHYDROLYSYS STEP

Table 5 hereunder sets out prehydrolysis conditions and results.

Table 5

Timber	Run No.	SO_2 charge (%)	L/W ratio	Time to temp (mins)	Time at Temp (mins)	Spent Liq. pH	Yield (%)
Gum	P6	0,2	5,3/1	60	120 @ 150°C	2,49	83,36
Gum	P7	0,2	5,3/1	60	120 @ 150°C	2,66	83,60
Gum	P8	0,2	5,3/1	60	120 @ 150°C	2,64	83,91
Gum	P9	0,2	5,3/1	60	120 @ 150°C	2,67	83,52
Gum	P10	0,2	5,3/1	60	120 @ 150°C	2,52	82,70
Wattle	P11	0,2	5,3/1	60	120 @ 150°C	2,78	79,32

The spent hydrolysis liquor from run no. P8 was titrated against standard NaOH solution in order to get an indication of the caustic soda consumption of such liquor. This would be relevant in the case where the prehydrolysis step is done in the same vessel or digester as the pulping step and the acidity of the liquor remaining after draining of the bulk of the prehydrolysis liquor has to be neutralized when adding pulping chemicals. Neutralization required 2,4g NaOH per litre of prehydrolysis liquor.

PULPING STEP

Pulping conditions and results are given in Table 6 hereunder.

The initial concentration of Na_2SO_3 (as NA_2O) at a charge of 17% at a L/W ratio of 3,5/l was 48,6 g/l. In order to reduce the Kappa number, cook number C5 was done at the same initial concentration as the other cooks but at a much higher charge (-L/W ratio increased to 5,3/l). The anthraquinone charge was also increased to 0,2% (on timber) and the time at top temperature was increased to 4 hours. The Kappa number could be lowered to 28,7 but this is still relatively high for a bleachable hardwood pulp. See hereunder under the heading "Extraction Step".

Table 6

Cook No.	Prehydrolysis No.	Chemical Charge (%)			L/W ratio	Time		Yield (%)		Kappa No.	Spent Liquor	
		Na_2SO_3 as Na_2O	Na_2CO_3 as Na_2O	A.Q.		To 175°C minutes	At 175°C hours	Screened	Rej.		Na_2SO_3 as Na_2O (g/l)	pH
C4	P6	17	4	0,15	3,5/1	80	3	53,02	0,02	40,6	11,32	9,21
C5	P6	25,74	6,075	0,2	5,3/1	80	4	49,02	0,01	28,7	19,43	9,16
C6	P7	17	4	0,15	3,5/1	80	3	53,26	0,01	37,8	10,73	9,33
C7	P8	17	4	0,15	3,5/1	80	3	53,23	0,01	34,1	10,28	9,21
C8	P9	17	4	0,1	3,5/1	80	3	53,63	0,02	37,4	9,45	8,84
C9	P10	17	4	0,1	3,5/1	80	3	53,74	0,03	41,1	10,33	9,10
C10	P11	17	4	0,1	3,5/1	80	1,5	57,94	0,50	31,7	15,15	9,0

THE EXTRACTION STEP

Table 7

Extraction No.	E5	E6	E7	E8	E9	E10
Cook No.	C4	C5	C6	-C7	C8+C9	C10
Initial Kappa No.	40,5	28,7	37,8	34,1	38,7	31,7
NaOH (%)	3	3	3	3	3	3
Temperature (°C)	70	70	70	70	70	70
Consistency (%)	10	10	10	10	10	10
Retention (hr)	1	1	1	1	1	1
Final pH	12,20	12,44	12,37	12,50	12,34	12,30
Kappa No.	14,5	12,4	15,2	14,0	20,3	16,0
Yield (%)	93,73	N.D.	94,93	95,31	95,86	96,79
Viscosity (cm^3/g)	998,4	-	1042,4	1095,9	1025,7	1184,8
D.P.	1500	-	1573	1663	1545	1812

The Kappa number drop during the extraction step was such that it was obviously more advantageous to use such a step than to try to reduce the Kappa number by using a more severe pulping step. This is clearly shown by comparing E5 and E6 in Table 7.

SINGLE VESSEL PROCEDURE

Cook number C11 (-see below) was done in order to simulate the procedure when doing prehydrolysis, pulping and the extraction step(s) in a single vessel (- batch digester, no washing between steps), as follows:

PREHYDROLYSIS STEP :

Wood charge	- 1400 g O.D.
L/W ratio	- 5,3/1
SO_2 (%)	- 0,2
Time to 150°C (mins)	- 60
Time at 150°C (hrs)	- 2
Liquor drained (ml)	- 5500
Liquor remaining (ml/calculated)	- 1920
Spent liquor pH	- 2,66

PULPING STEP :

Assumed prehydrolysis yield (%)	- 84
O.D. timber left (g)	- 1176
Na_2SO_3 charge (% Na_2O)	- 17%
Na_2CO_3 charge (% Na_2O)	- 4%
NaOH charge for neutralization	
1,920 x 2,4 g	- 4,608 g
A.Q. (%)	- 0,15
L/W ratio	- 3,5/1
Time to 175°C (mins)	- 80
Time at 175°C (hours)	- 3
Spent liquor residual sulphite (g/l Na_2O)	- 9,05
Spent liquor pH	- 8,89
Spent liquor volume (ml)	- 1800
Spent liquor left (ml/calculated)	- 2316

The amount of NaOH necessary to compensate for the acidity left after draining of the excess prehydrolysis liquor, came to 0,3% (as Na_2O) on O.D. - prehydrolised wood material.

EXTRACTION STEP :

Spent pulping liquor from cook number C6 was titrated against NaOH in order to determine the amount of caustic soda necessary to raise the pH to 11 (the approximate pH at which all $NaHCO_3$ will be converted to Na_2CO_3). This figure came to 12g NaOH per litre of spent pulping liquor.

Assumed pulping yield (%)	- 53
O.D. material left (g)	- 623,28 (assumed)
NaOH (%)	- 3

NaOH for raising pH of remaining pulping liquor to 11
= 2,316 x 12 = 27,792 g = 4,46%

Consistency (%)	- 10
Time at 80°C (mins)	- 20
Final pH	- 11,66
Kappa no.	- 13,4
Yield % - Screened	- 42,59
- Rejects	- 0,02

The final Kappa number of 13,4 was in line with figures obtained doing the three steps separately with washing between each step. The NaOH necessary to raise the pH of the remaining pulping liquor to 11 came to about 4,5% on O.D. pulp.

OXYGEN DELIGNIFICATION

In order to further lower the Kappa number, four of the unbleached pulps were subjected to oxygen delignification. See Table 8 below.

Table 8

Oxygen bleach no.	01	02	03	04
Starting pulp no.	E8	C11	E9	E10
Starting Kappa No.	14,0	13,4	20,3	16,0
NaOH (%)	1	1	2	1,5
Mg as $MgSO_4$ (%)	0,05	Nil	Nil	Nil
Consistency (%)	10	10	10	10
Time to temp. (mins)	22	16	26	26
Time at temp. (mins)	30	30	30	30
Top temperature (°C)	110	115	118	115
O_2 pressure (kPa)	690	690	690	690
Spent liquor pH	9,66	9,68	9,57	9,88
Yield (%)	97,86	98,25	95,45	96,37
Viscosity (cm^3/g)	973,6	946,8	784,8	1037,8
D.P.	1459	1415	1150	1566
Kappa Number	6,6	5,1	3,6	4,2

These pulps delignified easily to low Kappa numbers during an oxygen delignification step.

BLEACHING

Some caustic soda extracted pulps and the four oxygen delignified pulps were subjected to full bleaching. See Table 9 below.

Table 9

Starting Pulp	Bleach No.2	Sequence	Brightness % "ISO"	Alpha Cellulose %	Yield %		Viscosity cm³/g
					Bleached	On timber	
E5	B3	C2,9-E2,0-D0,6	90,3	97,79	97,10	40,22	840
E7	B4	C3,04-E2,0-D0,6-H0,6	91,7	96,78	96,72	40,88	654
01	B5	C1,32-E1,16-D0,4-H0,6	91,8	96,57	98,11	40,87	561
02	B6	C1,02-E1,01-D0,4-H0,6	91,6	96,14	98,67	41,29	620
03	B7	C0,726-E0,863-D0,3-H0,6	93,3	97,93	98,93	40,39	467
04	B8	D0,316-E0,916-D0,3-H0,6	93,5	94,88	98,57	42,26	718

EXAMPLE 3

In order to further illustrate the effect of an alkali extraction step after the pulping step and its interaction with the prehydrolysis step, a separate set of experiments was done using a new sample of run-of-mill (SAICCOR) Eucalyptus chips.

Prehydrolysis conditions were varied keeping L/W ratio constant at 5,0/l and temperature rise time at 1 hour. (Table 10)

Table 10

Prehydrolysis No	Hydrolysis agent (% on wood)	Temp. (°C)	Time at temp (mins)	Yield (%)	Effluent pH
P12	0,2 SO_2	150	120	84,63	2,71
P13	0,3 SO_2	160	150	78,11	2,50
P14	0,4 SO_2	170	180	73,34	2,37
P15	0,4 SO_2	150	120	77,14	2,30
P16	Pure water	150	120	88,53	3,24
P17	0,2 SO_2	150	60	90,08	2,74

Hydrolysed material was soaked for 30 minutes in water after treatment before washing and spin drying. Material originating from some of the prehydrolysis runs was subjected to pulping. (Table 11)

The following process conditions applied except for C17

3,5/l L/W ratio
0,1% Anthraquinone
16% Na_2SO_3 as Na_2O
4% Na_2CO_3 as Na_2O
80 minutes to temperature (175°C)
3 hours at 175°C

C17 was done using original wood material which had not been subjected to a prehydrolysis step. In this case the chemical charge was increased to 18% Na_2SO_3 (as Na_2O) and 4,5% Na_2CO_3 (as Na_2O) keeping the anthraquinone charge and other conditions as above.

Table 11

Cook No.	Pre-hydrolysis No.	Yield (%)		Spent Liquor Res. Sulphite as Na_2O (g/l)	pH	Kappa No
		Screened	Rejects			
C12	P12	51,72	1,04	3,07	8,83	35,4
C13	P13	52,11	0,80	15,24	8,88	75,3
C14	P14	50,61	5,81	16,43	8,89	170,1
C15	P15	53,45	3,60	14,46	8,86	93,7
C16	P16	51,01	0,40	5,20	9,02	30,1
C17		49,08	11,02	13,48	9,16	38,8

It was obvious that the prehydrolysis conditions had a large influence on delignification response in the pulping step. Harsher prehydrolysis conditions lead to higher pulp Kappa numbers and higher levels of residual sulphite in the spent liquor indicating greater deactivation of the lignin. This lignin had lost some of its fibre bonding properties however as the rejects obtained from pulping the prenudrolysis material were much lower than the rejects obtained from pulping the unhydrolised material in spite of the latter's lower Kappa number compared to figures obtained from pulping some of the prehydrolised material.

The pulps obtained as above were subjected to an alkaline extraction step using sodium hydroxide as the alkali. Extraction was done in plastic bottles in a constant temperature water bath. (See Table 12)

The following constant process conditions applied :

60 minutes retention time
10% consistency
temperature 70°C

Table 12

Extraction No	Cook No	NaOH (%)	Effluent pH	Kappa No	Kappa No Drop	Yield %	
						Stage	Screened on O. wood
E11	C12	3	12,07	20,0	15,4	96,00	42,02
E12	C12	1	11,33	26,4	9,0	97,36	42,70
E13	C12	2	11,92	22,9	12,5	96,51	42,24
E14	C12	4	12,12	17,5	17,9	95,31	41,72
E15	C13	3	11,69	23,3	52,0	89,06	36,25
E16	C14	3	11,40	84,5	85,6	82,91	30,77
E17	C15	3	11,67	39,0	54,7	88,96	36,68
E18	C16	3	12,32	22,6	7,5	97,77	44,15
E19	C17	4	12,37	31,1	7,7	99,01	

Comparing Kappa numbers of E11, E12, E13 and E14 shows that the Kappa number drop during the extraction step is dependent on the amount of alkali charged. In a separate experiment 100g O.D. of prehydrolysis -S.A.S.-A.Q. gum pulp of 38,3 Kappa number was washed with 1 litre of a 3g/1 NaOH solution at 70°C on a Buchner funnel. After washing with water the Kappa number of the resulting pulp was 25 indicating that merely washing with alkaline wash water can also effect a substantial Kappa number drop.

Comparing other extraction step Kappa numbers in Table 12 also indicates that the extraction step brings widely varying pulping step Kappa numbers, originating from varying prehydrolysis conditions, much closer together. This is a positive result but yield figures indicate that large yield losses can result from too harsh a prehydrolysis step. This step, however, must be sufficiently harsh (pH, temperature and time) to effect adequate hemicellulose removal.

The Kappa number drop obtained from the unhydrolised pulp (Cook C17) on extraction also indicates the different nature of the lignin in this pulp compared to the lignin in pulp which had been hydrolised. If this material had been obtained by applying a hydrolysis step prior to the pulping step, the Kappa number drop on extraction would have been more pronounced considering the unextracted Kappa number of this pulp.

The above results were obtained from pulp which had been well washed after the pulping step in order to demonstrate the positive effect of an alkaline extraction step.

When running batch digesters using this process the preferred procedure would be to drain the digester after the prehydrolysis step of the prehydrolysis spent liquor. (This liquor would be used for recovery and beneficiation of products dissolved and extracted from the wood).

The pulping chemicals (Na_2SO_3, Na_2 CO_3, and Anthraquinone) would then be charged (possibly together with a small amount of NaOH to compensate for the residual acidity of the prehydrolysis step). After a penetration period and pulping time, the spent liquor would be pumped from the digester. Alkali (NaOH) would then be added to this liquor and reintroduced to the digester followed by circulation for a period. This would complete the cook.

The addition of alkali to the spent liquor and reintroduction of this mixture to the digester serves two purposes.

a) An effective extraction is accomplished which lowers the Kappa number prior to the oxygen delignification step (or other step depending on the bleach sequence). The pH adjusted spent liquor can be introduced at high temperature because the free alkali concentration is low which otherwise would have resulted in cellulose yield loss at high temperature. The results appear to indicate that the lowering of the Kappa number is mostly the result of fibre swelling at the higher pH. Pathways are opened as a result of the swelling which allow removal of some of the lignin molecules which are loosely held.

b) Spent S.A.S.-A.Q. pulping liquor can be difficult to evaporate to high solids levels without forming troublesome precipitates. This can be overcome by adjusting the pH of the spent liquor to about 11 prior to evaporation.

In a continuous digester introduction of pH adjusted spent liquor to the wash zone can accomplish both the extraction step Kappa reduction benefit and the enhanced liquor evaporation properties resulting from the higher pH.

It appears from the above that the process of the present invention can produce high alpha-cellulose pulp at a high pulp yield. An alkaline extraction step following pulping proved to be satisfactorily effective in removing residual lignin.

An oxygen bleaching step also proved effective in further lowering the Kappa number to acceptably low levels prior to full bleaching. Full bleaching of such pulps required fairly low charges of chlorine-containing chemicals, thereby providing a process which has a low pollution potential. "Non polluting" bleach chemicals like ozone and peroxide could also be considered.

Although certain embodiments only of the invention have been illustrated above, it will be appreciated by any person skilled in the art that other possibilities and variations of the invention are possible. Such possibilities and variations are therefore to be considered as falling within the spirit and scope of the present invention as defined in the appended claims.

Claims

1. A process for producing dissolving grade pulp, the process including -
   applying a prehydrolysing step to a lignocellulosic-containing material.
   partly dissolving the lignin contained in the prehydrolysed material (- a pulping step) ; and
   carrying out an alkali extraction step.

2. A process as claimed in claim 1 wherein about 2% to 5%, by mass of alkali is used to carry out the alkali extraction step.

3. A process as claimed in claim 2, wherein about 3%, by mass of alkali is used to carry out the alkali extraction step.

4. A process as claimed in claim 3, wherein the alkali used is sodium hydroxide.

5. A process as claimed in any one of the preceding claims, wherein the alkali extraction step is carried out at a temperature of about 50°C to 100°C for a period sufficient to give optimal lignin removal.

6. A process as claimed in claim 5, wherein the alkali extraction step is carried out at a temperature of about 70°C to 80°C for a period sufficient to give optimal lignin removal.

7. A process for reducing the lignin content (as expressed by the Kappa number) of unbleached pulp intended for dissolving grade pulp manufacture, the process including applying an alkali extraction step to the pulp.

8. A process as claimed in claim 7 wherein about 2% to 5%, by mass of alkali is used to carry out the alkali extraction step.

9. A process as claimed in claim 8, wherein about 3%, by mass of alkali is used to carry out the alkali extraction step.

10. A process as claimed in any one of claims 7 to 9, wherein the alkali used is sodium hydroxide.

11. A process as claimed in any one of claims 7 to 10, wherein the alkali extraction step is carried out at a temperature of about 50°C to 100°C for a period sufficient to give optimal lignin removal.

12. A process as claimed in claim 11, wherein the alkali extraction step is carried out at a temperature of about 70°C to 80°C for a period sufficient to give optimal lignin removal.

13. A process as claimed in any one of the preceding claims, wherein spent liquor from the pulping step is removed from a digester, treated with alkali, and re-introduced to the digester to lower the Kappa number of the pulp and/or to improve evaporation of the spent liquor.

14. A process as claimed in claim 13, wherein after re-introduction of the spent liquor to the digester, the digester is operated at a temperature > 100°C and at elevated pressure.

15. A process as claimed in either claim 13 or claim 14, wherein about 10% to 15% alkali by mass of pulp is used.

16. A process as claimed in any one of claims 13 to 15, wherein the alkali used is sodium hydroxide.

17. A process as claimed in either claim 1 or claim 7, substantially as herein described and/or exemplified.

18. A dissolving grade pulp whenever produced by a process as claimed in any one of the preceding claims.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 91 40 0355

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	ABSTRACT BULLETIN OF THE INSTITUTE OF PAPER CHEMISTRY. vol. 40, no. 4, October 1969, APPLETON US pages 292 - 293; Nepenin,Y.N. and Zhalina: "High-yield viscose-grade pulp." * abstract * ---	1, 4, 7, 10, 17, 18	D21C1/04 D21C3/26
X	ABSTRACT BULLETIN OF THE INSTITUTE OF PAPER CHEMISTRY. vol. 58, no. 1, July 1987, APPLETON US page 83 Petrik,A.W. et al.: "Production of dissolving pulp in a continuous digester using the Kraft process with hydrolysis." * abstract * ---	1, 7, 17, 18	
A	US-A-2301314 (UNIVERSITY OF MINNESOTA) * claim 1 * -----	1, 4, 7, 10, 17, 18	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			D21C

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	21 MAY 1991	BERNARDO NORIEGA F.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

EPO FORM 1503 03.82 (P0401)