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

(11) **EP 1 552 052 B1**

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention of the grant of the patent:
03.10.2007 Bulletin 2007/40

(21) Application number: **03794378.4**

(22) Date of filing: **21.08.2003**

(51) Int Cl.:
D21B 1/02 *(2006.01)*

(86) International application number:
PCT/SE2003/001296

(87) International publication number:
WO 2004/022842 (18.03.2004 Gazette 2004/12)

(54) **A METHOD OF PRODUCING MECHANICAL PULP AND THE MECHANICAL PULP THUS PRODUCED**

VERFAHREN ZUR HERSTELLUNG VON HOLZSTOFF UND DER SO HERGESTELLTE HOLZSTOFF

PROCEDE DE PRODUCTION DE PATE MECANIQUE ET PATE MECANIQUE AINSI PRODUITE

(84) Designated Contracting States:
AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LI LU MC NL PT RO SE SI SK TR
Designated Extension States:
LT LV

(30) Priority: **06.09.2002 SE 0202629**

(43) Date of publication of application:
13.07.2005 Bulletin 2005/28

(73) Proprietor: **Stora Enso Aktiebolag**
791 80 Falun (SE)

(72) Inventors:
- **PENG, Frank**
  S-663 41 HAMMARÖ (SE)
- **FERRITSIUS, Rita**
  S-791 33 FALUN (SE)
- **ÄNGSAS, Ulf**
  S-791 74 FALUN (SE)

(74) Representative: **Johansson, Lars-Erik et al**
Hynell Patenttjänst AB
Patron Carls väg 2
683 40 Hagfors / Uddeholm (SE)

(56) References cited:
EP-A1- 0 430 915
WO-A1-89/02951
WO-A1-92/20855
WO-A1-98/06892
US-A- 4 891 096

- **ZHUNG ZHONG LEE ET AL.: 'Pectic substances contribute to fiber-fiber bonding' TAPPI vol. 65, no. 2, February 1982, pages 61 - 63, XP002974395**

EP 1 552 052 B1

Printed by Jouve, 75001 PARIS (FR)

Description

TECHNICAL FIELD

[0001] The present invention relates to a method of mechanical pulping based on cellulose fiber material after pretreatment of the fiber material with pectinase prior to mechanical defibration. The invention is also related to the mechanical pulp thus produced.

PRIOR ART

[0002] In mechanical pulping wood fibers are separated by mechanical defibration, typically in a refiner or a grinder, with or without pretreatment. When pretreatment is applied, the process can be either TMP, where thermal softening is applied; or CTMP, where for instance sulfite is used for the sulfonation of lignin; or APMP, where alkaline peroxide is applied resulting in a combined effect of chemical softening due to alkali and bleaching or brightening due to peroxide. In almost all the mechanical pulping processes, thermal treatment is employed prior to refining to soften the wood material thus leading to reduced energy consumption as well as improved pulp strength. While alkaline treatment is an effective treatment for reducing energy consumption, it also induces darkening reactions that result in poorer optical properties that are essential for paper products based on mechanical pulps. Besides, the use of alkali results in extensive dissolution of wood material, which results in yield loss and necessitates effluent treatment.

[0003] In addition to conventional processes, there have been efforts to apply biotechnology to mechanical pulping. In WO 97/40194, a mechanical pulping process using various fungi or enzymes is described. The proposed treatment includes compression of wood chips followed by impregnation with a biological agent prior to refiner treatment. Although a great number of fungi and enzymes are referred to in the patent application, the biological agents used in the given examples are limited either to fungus with ligninase activity, or to enzymes based on xylanase activity aiming for the hemicellulose, and lipase activity specific for the wood resin. The examples show that the enzymatic treatment can reduce the refiner energy by 4 to 10%, in addition to the energy reduction of about 4% due to the compression treatment as compared to the reference. At the same time the strength properties of the pulp are improved when compared at the same pulp freeness.

[0004] Another biological pulping process involving pretreatment of wood chips is based on lignin oxidizing fungi treatment (US 5,055,159). The refining energy consumption can be reduced by about 30% at the same freeness. The treatment however requires long incubation time and the pulp produced has a dark color.

[0005] Different to the above-mentioned applications, pectinase has been specifically used in post-treatment of mechanical pulps. In US 5,487,812 pectinase is used for alkaline bleached mechanical wood pulp. The purpose of such treatment is however to remove pectin remaining after the pulping process in order to reduce the source of so-called "anionic trash" which is harmful in paper machine operation.

[0006] EP-A-0 430 915 relates to enzymes for decomposition of hemicellulose and/or cellulose, in order to achieve a reduction in energy consumption in the production of mechanical pulp. The example given shows the efficiency of hemicellulase in reduction of refining energy in the treatment of a coarsely separated pulp. However, no results are shown for the suggested use of pectinase.

[0007] WO-A-89/02951 teaches how to treat wood pieces having a length in the fibre direction of at least 100 mm, in order to achieve a better pulp strength due to fibre separation. From the document, the skilled person can learn that compression of at least 5 MPa has such an effect, but that the method is difficult to employ generally on chips that are of considerably smaller size and that moreover are more or less randomly oriented at compression.

SUMMARY OF THE INVENTION

[0008] The present invention aims at providing a method of mechanical pulping requiring significantly reduced refining energy consumption to reach a certain pulp freeness, or pulp strength, as compared to prior art. The invention also aims at improved optical properties in the pulp, such as an improved brightness.

[0009] This is accomplished by the method according to the invention, as defined in the claims.

[0010] According to the invention, the method comprises pretreatment of the comminuted cellulose fiber material with pectinase, alone or in combination with a chelating agent, for instance DTPA and/or sulfite, followed by mechanical defibration and refining to produce a mechanical pulp.

[0011] Pectin is a group of amorphous polysaccharide substances in wood. Although the amount of pectin in softwood and hardwood is normally less than 1%, it is predominantly deposited in the compound middle lamella, and the tori of bordered pit-membranes of coniferous species (Hafren J. and Westermark U.: Nordic Pulp and Paper 16 (4), 284-290, 2001). The main component of pectin is polygalacturonan consisting of galacturonic acid units that to various extend are esterified. When in free acid form, the carboxylic groups of the galacturonan having a negative charge can induce

local swelling of the fibers in contact with aqueous solutions. The esterified groups, on the other hand, can be de-esterified through for instance alkaline treatment and thus contributing to swelling.

[0012] Given the technical background, the present invention is aimed to utilize the specific features of pectin with respect to its specific location and the potential to induce selective weakening in the fiber wall. Enzyme with specific activity towards pectin is used for the pretreatment of cellulose fiber material. Maceration of the fiber material prior to the enzyme treatment proves an efficient way to get the enzyme in place. To render the fiber separation in the region where chemical weakening occurs, the refining condition is optimized in respect to refining intensity and preheating temperature. The results according to the present invention are a significantly reduced refining energy to reach a given pulp freeness and improved strength and optical properties of the pulp.

[0013] According to one aspect of the invention, the pretreatment is preferably assisted by a mechanical compression to facilitate liquor uptake and to give a more even treatment of the material. Compression screw device or twin roll presses can be used at a compression ratio of 1:1 to 8:1. The pretreatment can be further improved if presteaming is conducted prior to the compression.

[0014] Impregnation according to the invention follows immediately after the compression and/or thermal pretreatment. The pectinase is charged in an amount required for sufficient treatment, preferably 2,000,000 to 200,000,000 polygalacturonase units/ton material and even more preferred 10,000,000 to 50,000,000 polygalacturonase units/ton. The retention time of treatment is 3 min to 24 hours, preferably 3 to 300 min, even more preferred 15 to 240 min and most preferred 30 to 120 min at a temperature of 20 to 100°C, preferably 35 to 70°C, and more preferably about 50°C. The treatment condition can be adjusted depending on the charge of the pectinase to allow for a sufficient hydrolysis of the pectins. Other pectinase preparations can be used provided that the pectolytic activity is satisfied.

[0015] Defibration and refining of the pectinase treated material can be carried out either at conventional TMP conditions and high intensity conditions. A surprising energy reduction effect is obtained. To reach a given pulp freeness of 100 ml CSF, the energy reduction is 400 kWh/t with TMP condition, from 2500 kWh/t without pectinase treatment to 2100 kWh/t with pectinase treatment, or by about 16%. The energy reduction is 150 kWh/t with high intensity condition, from 2150 kWh/t without pectinase treatment to 2000 kWh/t with pectinase treatment, or by about 7%. The combination of high intensity and pectinase treatment gives a total reduction of 500 kWh/t, from 2500 kWh/t to 2000 kWh/t at a pulp freeness of 100 ml CSF, which is 20% in reduction. The shives content decreases more rapidly after the pectinase treatment.

[0016] The strength properties remain the same as compared to conventional TMP, and improved by about 10% as compared to high intensity TMP. The brightness of the pulp is also increased by the enzymatic treatment.

[0017] While the addition of DTPA or sulfite doesn't contribute to further energy reduction, it increases the brightness by 2 to 5 brightness units. The improved brightness is maintained after peroxide bleaching, together with the benefit of an increased amount of residual peroxide for recycling.

[0018] The surprising effects resulting from the pectinase treatment could be explained by a selective weakening of the pectin-enriched region in the fiber wall due to hydrolysis of the pectins, with the consequence of a more efficient fiber separation in defibration and refining of the material. It is evident to those skilled in the art that this process can bring about a significant economical benefit, in terms of improved pulp properties and reduced energy cost. It is also evident to those skilled in the art that any enzyme preparation that contains sufficient pectolytic hydrolysis activity, alone or in combination with other chemicals, can be used for the treatment according to the present invention.

The present invention describes a mechanical pulping process comprising:

DETAILED DESCRIPTION OF THE INVENTION

[0019]

A) Mechanical pretreatment of the comminuted fiber material with suitable mechanical means, for instance compression with screw device or roll press device, to enable an efficient uptake of aqueous solution into the fiber material during an impregnation immediately following the pretreatment. Replacement of resin-containing liquor in natural cellulose fiber material with added aqueous solution is also positive in a process point of view. The compression ratio is preferably in the range of 1:1 to 8:1, preferably 2:1 to 5:1, where 1:1 means a screw conveyer transporting the fiber material into the impregnation bin. In combination with compression treatment, an initial thermal heating of the fiber material may further facilitate an efficient uptake of the aqueous solution, preferably by use of fresh or recycled steam at atmospheric pressure, for 1 to 30 min, preferably 10 to 20 min. As an alternative, thermal heating followed immediately by impregnation may be used instead of compression treatment when no such facility is available. The pretreatment procedure can also be repeated in one to several stages with the freedom of applying various impregnation chemicals in different stages.

B) Impregnation of the fiber material with pectinase-containing aqueous liquid. Added in connection with or immediately after the pretreatment stage, the enzyme can more easily reach the sites of reaction. In addition to the

pectinase enzyme, other chemicals for instance chelating agents such as sulfite, preferably at a charge of 5 to 50 kg/ton, and/or DTPA, preferably at a charge of 1 to 10 kg/ton, may be added in the impregnation to further improve the process. The pectinase-containing liquid may comprise an enzymatic preparation with pectolytic activity for both un-esterified pectins and esterified pectins. Moreover, the aqueous liquid may comprise two or more enzymatic preparations wherein at least one of the preparations has pectinase activity; or the pectinase may be added as a biological agent comprising one or more fungi or bacteria, at least one of which having pectolytic activity. The charge of enzyme is subject to process conditions and cost effectiveness, and lies in the range of 2,000,000 to 200,000,000 polygalacturonase units/ton fiber material, preferably 10,000,000 to 50,000,000 polygalacturonase units/ton. The pH of the impregnation liquor is adjusted by alkali or acid, preferably by caustic soda or mineral acid, to a value optimal for enzymatic reactions, suitably in the pH range of 3 to 10, preferably 4 to 7 and more preferably about 5. The retention time and temperature are also adjusted depending on the process setup and reaction requirements, preferably being 3 to 300 min at a temperature of 20 to 100°C. It is evident to those skilled in the art that any enzyme that contains sufficient pectolytic activity, alone or in combination with other chemicals, can be used in the treatment as described.

C) Defibration and refining of the impregnated fiber material is carried out under optimized conditions. The fiber material is preferably preheated prior to feeding into the pressurized primary refiner, to allow for thermal softening of the fiber wall. The pulping conditions can comprise a refiner rotation speed of 1000 to 3000 rpm, preferably 1500 to 2600 rpm, using either conventional TMP or high refining intensity. Conventional TMP conditions may comprise preheating at 0 and up to 4 or 5 bar with a retention time of 2-10 min in the preheater and a refiner rotation speed of 1200 to 1800 rpm. The pressure 0 bar means atmospheric refining. High intensity conditions may comprise preheating at above 4 or 5 bar and up to 8 bar with a retention time of 3 to 30 sec and a refiner rotation speed above 2000 rpm but usually not above 3000 rpm. The retention time should be matched against the preheating temperature (steam pressure) as high preheating temperature requires shorter retention time. After the primary refining, a secondary refining stage can be used to reach the required pulp freeness. The second-dary refining stage may have the same conditions as the primary stage.

[0020] Although disc refiners are the defibration and refining equipment used in this study, other mechanical devices can be used for the purpose of defibration and refining. Some of the examples are conical refiners and stone ground wood grinders. The refiners can be of the single disc or double disc type. The refining may be made in a single stage or in multistage. The defibration and refining can also be done at atmospheric pressure, in which case refiner mechanical pulping, or RMP is a more appropriate description of the process instead of TMP.

[0021] Although only softwood fiber material is used in this study, any fiber material containing pectins could be treated with this method. The fiber material could be hardwood fiber material, non-wood fibers such as bagasse, bamboo, reed and straw.

[0022] The embodiment as described here can be modified according to the source of fiber material and the process set-up.

[0023] The pulp obtained can depending on the application of the pulp be subject to further treatment such as washing, screening, post-refining and bleaching according to conventional processes.

BRIEF DESCRIPTION OF THE DRAWINGS

[0024] In the following, there will be given a few examples of preferred embodiments according to the invention. The invention is however not limited to these examples. Reference is made to the enclosed drawing diagrams, of which:

Fig. 1 is showing freeness vs. specific refining energy for Example 1,
Fig. 2 is showing freeness vs. specific refining energy for Example 2,
Fig. 3 is showing freeness vs. specific refining energy for Example 3.

EXAMPLES

[0025] Mechanical pulps are produced by different chemical treatments, in a pilot refiner plant. The process conditions are as follows.

Material

[0026] Wood chips consisting of approximately one third white spruce, one third red and black spruce and one third balsam fir, all of Canadian origin in the Nova Scotia region. The chips are prepared with mill-size chipper and well mixed

prior to the trials.

Pretreatment

[0027] The chips are pre-steamed at atmospheric pressure for 15 min. After pre-steaming the chips are fed into a plug-screw device with a compression ratio of 4:1. At the outlet of the device, the chips are released into a solution containing various chemicals, including pectinase in the trials according to the invention. The pH of the solution is kept at about 5 adjusted by use of sodium hydroxide or sulfuric acid.

Impregnation and retention

[0028] After passing through the impregnation bin, the chips are stored in well-isolated barrels. The retention time is 120 min at a temperature around 50°C. The pectinase used in this study is a highly pectolytic enzyme preparation. This enzyme also hydrolyzes the methyl-esterified galacturonic acid in the pectins. The pectinase used is generated by submerged fermentation of a group of microorganisms containing *Aspergillus aculeatus* and *Aspergillus oryzae*. The charge of pectinase in this study is 30,800,000 polygalacturonase units/ton. The charges of DTPA and sodium sulfite are 4 kg/t and 12 kg/t, respectively, were applicable.

Defibration and refining

[0029] The primary refiner is of the Sprout-Bauer 36-1 CP single disc refiner type. Two pulping conditions, conventional TMP and high intensity HI-TMP, are applied. The preheating steam pressure is 2.8 bar for TMP and 5.9 bar for high intensity (HI-TMP). The retention time at preheating is 3.4 min for TMP and 12 sec for HI-TMP. The refiner rotation speed is 1800 rpm for TMP and 2600 rpm for HI-TMP. The secondary refining is carried out with a double disc atmospheric refiner to reach the final freeness levels.

[0030] The obtained pulps are evaluated both for strength properties and optical properties including a bleachability study with hydrogen peroxide bleaching.

EXAMPLE 1

[0031] In this trial TMP conditions are applied for both samples with the difference that reference, Sample 1, is impregnated only with water adjusted to pH 5.

[0032] Sample 1: TMP condition with only water impregnation with pH adjusted to 5.

[0033] Sample 2: TMP condition with 30,800,000 polygalacturonase units/t pectinase, pH 5.

[0034] It is shown in Table 1 that the specific energy consumption required to reach a given freeness is significantly reduced by the enzymatic treatment. The degree of reduction is approximately 400 kWh/t at a given freeness, which also can be seen in Figure 1. The pulp properties for those samples are comparable to each other over the freeness range applicable for paper manufacture. The shives content is more rapidly reduced by the pectinase treatment. At a freeness of 57 ml CSF, the shives content is 0.12% as compared to the reference, 0.34%.

Table 1.

	Sample 1					Sample 2			
Freeness, ml CSF	665	230	148	129	57	656	115	57	36
SEC (kWh/t)	902	1889	2028	2237	3078	824	1993	2386	3000
Density (kg/m3)		309,6	344,83	371,75	440,53	205,76	384,62	446,43	462,96
Bulk (cm3/g)		3,23	2,9	2,69	2,27	4,86	2,6	2,24	2,16
Burst ind. (kPam2/g)		1,65	1,98	2,29	3,04	0,34	2,46	3,1	3,35
Tear ind. (mNm2/g)		10,9	9,6	9,5	7,9	4,8	8,7	8	7,4
Tens ind. (N.m/g)		33,9	39,9	42,7	53,5	10,1	42,9	55,5	57,4
%Stretch		2,03	2	2,08	2,57	1,1	2,2	2,5	2,67
T.E.A.(J/m2)		28,48	32,7	36,95	57,6	4,6	39,7	28,6	64,3
%Opacity		97	97	97,4	98,4	88,2	98,1	97,5	97,5
Light scatt. coe.(m2/kg)		50,1	52,1	52,8	61,4	35,3	53,9	59,2	65,1

(continued)

	Sample 1					Sample 2			
ISO%	44,5	48,8	48,6	49,8	62,3	46,6	50	52,6	52,2
%shives(Pulmac-0.1mm)	15	1,3	0,98	0,8	0,34	13,9	0,6	0,12	0,05

EXAMPLE 2

[0035] In this trial the high intensity condition is applied for both samples with the difference that the reference, Sample 3, is impregnated only with water adjusted to pH 5.

[0036] Sample 3: HI-TMP condition with only water in the impregnation adjusted to pH 5.

[0037] Sample 4: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, pH 5.

Table 2.

	Sample 3					Sample 4				
Freeness, ml CSF	714	293	197	126	54	725	254	236	121	72
SEC (kWh/t)	531	1482	1652	1897	2655	412	1362	1462	1793	2344
Density(kg/m3)		311,53	362,32	389,11	454,55		317,46	321,54	387,6	423,73
Bulk (cm3/g)		3,21	2,76	2,57	2,2		3,15	3,11	2,58	2,36
Burst ind. (kPa.m2/g)		1,47	1,78	2,07	2,96		1,82	1,85	2,6	3,2
Tearind. (mNm2/g)		10,1	8,3	8,5	7,7		11,1	11,1	9,4	9,2
Tens ind. (N.m/g)		30,2	34,9	40,8	51,6		34,7	35,4	47,3	53,3
%Stretch		1,97	2,08	2,03	2,63		2	2,1	2,5	2,54
T.EA.(J/m2)		24,73	29,74	35,27	58,53		30,1	32,4	49,3	57,3
%Opacity		94,6	95,4	96,7	97,2		95,3	94,8	96,6	97,1
Light scatt.coe. (m2/kg)		41,2	49,3	61,6	62,6		49,4	48,9	59,3	60,8
ISO%	45,3	50,3	50,1	52	53,9	44,8	50,6	50,8	51,6	54,4
%shives (Pulmac-0.1mm)	8,42	0,62	0,5	0,38	0,04	8,76	0,52	0,4	0,14	0,06

[0038] It is shown in Table 2 and Figure 2 that the enzymatic treatment has a surprising effect on energy reduction also with the high intensity condition. To reach a given freeness of down to about 100 ml CSF, the specific energy consumption is reduced by about 150 kWh/t. The reduction is less pronounced at lower freeness. The pulp properties at the same time are improved by about 10% both for tensile strength and tear strength, while the optical properties remain at the same level. The potential of the enzymatic treatment makes is obvious to people skilled in the art that even better strength can be achieved if the same amount of energy is applied to the enzymatically treated pulps, or even less energy is required if the same strength properties are to be acquired.

EXAMPLE 3

[0039] In this trial the HI-TMP condition is applied for all the samples with Sample 3 and Sample 4 from Example 2 as references.

[0040] Sample 3: HI-TMP condition with only water in the impregnation adjusted to pH 5.

[0041] Sample 4: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, pH 5.

[0042] Sample 5: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, 4 kg/t DTPA, pH 5.

[0043] Sample 6: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, 4 kg/t DTPA and 12 kg/t sodium sulfite, pH 5.

Table 3

	Sample 3				Sample 4				Sample 5				Sample 6			
Freeness, ml CSF	293	197	126	54	254	236	121	72	201	148	87	52	184	150	97	55
SEC (kWh/t)	1482	1652	1897	2655	1362	1462	1793	2344	1539	1681	2022	2676	1934	2082	2303	3040
Density (kg/m3)	311,53	362,32	389,11	454,55	317,46	321,54	387,6	423,73	340,14	362,32	413,22	446,43	384,62	358,42	398,41	436,68
Bulk (cm3/g)	3,21	2,76	2,57	2,2	3,15	3,11	2,58	2,36	2,94	2,76	2,42	2,24	2,6	2,79	2,51	2,29
Burst ind. (kPa.m2/g)	1,47	1,78	2,07	2,96	1,82	1,85	2,6	3,2	1,87	2	2,63	3,1	2,3	2,24	2,61	3
Tear ind. (mNm2/g)	10,1	8,3	8,5	7,7	11,1	11,1	9,4	9,2	8,6	8,8	8,5	7,8	9,8	9,5	10,3	7,4
Tens ind. (N.m/g)	30,2	34,9	40,8	51,6	34,7	35,4	47,3	53,3	37,1	41,3	47,1	52,8	41,7	41,3	47,1	51,1
%Stretch	1,97	2,08	2,03	2,63	2	2,1	2,5	2,54	2,2	2,1	2,56	2,7	2,1	2,5	2,6	2,9
T.E.A. (J/m2)	24,73	29,74	35,27	58,53	30,1	32,4	49,3	57,3	33	37,9	48,3	57	35,2	40,1	47,2	56,6
%Opacity	94,6	95,4	96,7	97,2	95,3	94,8	96,6	97,1	94,1	95,4	96	96,6	93,2	94,2	95,2	95,5
Light scatt.coe. (m2/kg)	41,2	49,3	61,6	62,6	49,4	48,9	59,3	60,8	52,5	55,6	58,2	60,2	49,3	53,6	57,8	60,1
ISO%	50,3	50,1	52	53,9	50,6	50,8	51,6	54,4	52,1	52,9	54	56	55,4	55,8	56,3	58,8
%shives (Pulmac-0.1mm)	0,62	0,5	0,38	0,04	0,52	0,4	0,14	0,06	0,68	0,54	0,3	0,07	1	0,68	0,48	0,09

[0044] The refining energy consumption, SEC, is not affected by additional charge of DTPA or DTPA/sulfite. The SEC is at the same level for all samples with pectinase treatment, about 150 kWh/t lower compared to Sample 3, i.e. high intensity condition without pectinase treatment, also see Figure 3. A surprising effect is however obtained in the brightness of unbleached pulp. With DTPA together with pectinase, the brightness is increased by about 2 units ISO. With a combined of pectinase, DTPA and sulfite, the brightness improvement is even higher, 4-5 units ISO. The brightness improvement is fairly constant in the entire freeness range.

EXAMPLE 4

[0045] Pulps with different treatments are bleached with alkaline peroxide with following bleach conditions:

- Pretreatment with 7 kg DTPA/t at 7% consistency, 90°C, pH 5 for 45 min before dewatering to 30%.
- Bleaching at 25% consistency at 30kg/t hydrogen peroxide charge, 80°C for 90 min. Three sodium hydroxide charges, 10, 20 and 30kg/t, are used to find the optimal brightness. The optimal brightness and corresponding residual peroxide are given as bleach results.

[0046] Sample 7: TMP condition with only water impregnation with pH adjusted to 5.
[0047] Sample 8: TMP condition with 30,800,000 polygalacturonase units/t pectinase, pH 5.
[0048] Sample 9: HI-TMP condition with only water in the impregnation adjusted to pH 5.
[0049] Sample 10: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, pH 5.
[0050] Sample 11: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, 4 kg/t DTPA, pH 5.
[0051] Sample 12: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, 4 kg/t DTPA and 12 kg/t sodium sulfite, pH 5.

Table 4

	Unbl. ISO%	Bleached ISO%	Res. H2O2 %
Sample 7	52,3	68,1	7,5
Sample 8	54,7	70,6	16,8
Sample 9	53,9	68,3	5,6
Sample 10	54,4	71,9	19,9
Sample 11	56.0	71,4	12,9
Sample 12	59,5	74,3	25,3

[0052] It is shown in Table 4 that the brightness improvement is maintained after the enzymatic treatment. The improvement is about 2.5 brightness units for the TMP condition, and 3.6 units for the HI-TMP condition. Combination of DTPA and sulfite in the impregnation gives further improvement to a brightness of 74.3%ISO, compared to the TMP reference of 68.1%ISO.
[0053] Noticeably, the residual peroxide is much higher with the enzymatically treated samples. This is beneficial because the residual chemicals are recycled back to the process after bleaching. The need of make-up peroxide is reduced accordingly.
[0054] The invention is not limited by the embodiments described above, but may be varied within the scope of the claims.

Claims

1. A method of producing mechanical pulp wherein, for reducing refining energy consumption while improving optical and strength properties of the produced mechanical pulp, the method comprises the steps of:

   a) initially compressing a fiber material;
   b) selectively weakening a pectin-enriched region in the fiber walls of the fiber material by impregnating the fiber material with a pectinase-containing aqueous liquid, resulting in hydrolysis of the pectins;
   c) defibrating and refining the fiber material to produce a mechanical pulp.

2. A method according to claim 1, **characterised in that** said initial compression of the fiber material is a mechanical compression, preferably combined with a thermal pretreatment of the fiber material, preferably by steaming, before

the impregnation.

3. A method according to claim 2, **characterised in that** steaming is carried out, preferably at atmospheric pressure, for 1 to 30 min, preferably 10 to 20 min.

4. A method according to claim 2 or 3, **characterised in that** compression is performed by a compression screw or a twin roll press, with a compression ratio of 1:1 to 8:1, preferably 2:1 to 5:1.

5. A method according to anyone of the preceding claims, **characterised in that** the pectinase-containing liquid comprises an enzymatic preparation with pectolytic activity for both pectins and esterified pectins.

6. A method according to anyone of the preceding claims, **characterised in that** the aqueous liquid comprises two or more enzymatic preparations wherein at least one of the preparations has pectinase activity.

7. A method according to anyone of claims 1-5, **characterised in that** the pectinase is added as a biological agent comprising one or more fungi or bacteria, at least one of which having pectolytic activity.

8. A method according to anyone of the preceding claims, **characterised in that** the pectinase arises from a group of microorganisms containing *Aspergillus aculeatus* and *Aspergillus oryzae*.

9. A method according to anyone of the preceding claims, **characterised in that** the charge of pectinase is 2,000,000 to 200,000,000 polygalacturonase units/ton fiber material, preferably 10,000,000 to 50,000,000 polygalacturonase units/ton.

10. A method according to anyone of the preceding claims, **characterised in that** the aqueous liquid comprises at least one chelating agent, preferably diethylenetetraminepentaacetic acid at a charge of 1 to 10 kg/ton and/or sulfite at a charge of 5 to 50 kg/ton.

11. A method according to anyone of the preceding claims, **characterised in that** a retention time after uptake of the impregnation liquid is 3 min to 24 hours, preferably 15 to 240 min, and more preferably 30 to 120 min.

12. A method according to claim 11, **characterised in that** a temperature in the retention after uptake of the impregnation liquid is 20 to 100°C, preferably 35 to 70°C, and more preferably about 50°C.

13. A method according to anyone of the preceding claims, **characterised in that** a pH in the impregnation liquid is 3 to 10, preferably 4 to 7, and more preferably about 5.

14. A method according to anyone of the preceding claims, **characterised in that** the defibration and refining of the fiber material is performed by use of single disc, double disc or conical refiners in one or multi stages.

15. A method according to claim 14, **characterised in that** a refiner rotation speed is 1000 to 3000 rpm, preferably 1500 to 2600 rpm.

16. A method according to claim 14, **characterised in that** the fiber material is preheated for 2 to 10 min before refining, that a refiner pressure is from atmospheric up to 5 bar, preferably up to 4 bar and that a refiner rotation speed preferably is 1200 to 1800 rpm.

17. A method according to claim 14, **characterised in that** the fiber material is preheated for 3 to 30 sec before refining, that a refiner pressure is from 4 to 8 bar, preferably 5 to 8 bar and that a refiner rotation speed preferably is above 2000 rpm.

18. A method according to anyone of the preceding claims, **characterised in that** said fiber material is softwood chips or hardwood chips.

19. A method according to anyone of claims 1-17, **characterised in that** the fiber material is non-wood fiber material including bagasse, bamboo, reed and straw.

20. A method according to anyone of the preceding claims, **characterised in that** the pulp obtained after defibration

and refining is bleached, preferably with alkaline peroxide, to obtain bleached pulp having high brightness.

21. Mechanical pulp, **characterised in that** it has been produced by a method according to any one of claims 1-20.

Patentansprüche

1. Verfahren zur Herstellung von Holzstoff, bei welchem zur Verringerung des Energieverbrauchs bei der Mahlung unter Verbesserung der optischen Eigenschaften und des Festigkeitsverhaltens des so hergestellten Holzstoffs das Verfahren die folgenden Schritte umfasst:

a) anfängliches Verdichten eines Fasermaterials;
b) selektive Verringerung der Festigkeit eines mit Pektin angereicherten Bereichs in den Faserwänden des Fasermaterials durch Imprägnieren des Fasermaterials mit einer Pektinase enthaltenden wässrigen Flüssigkeit, was zur Hydrolyse der Pektine führt;
c) Zerfasern und Mahlen des Fasermaterials zur Bildung eines Holzstoffs.

2. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, dass** es sich bei der anfänglichen Verdichtung des Fasermaterials um eine mechanische Verdichtung handelt, vorzugsweise in Verbindung mit einer Wärme-Vorbehandlung des Fasermaterials, vorzugsweise durch Dampfbehandlung, vor der Imprägnierung.

3. Verfahren nach Anspruch 2, **dadurch gekennzeichnet, dass** die Dampfbehandlung vorzugsweise bei atmosphärischem Druck 1 bis 30 Minuten lang, vorzugsweise 10 bis 20 Minuten lang, durchgeführt wird.

4. Verfahren nach Anspruch 2 oder 3, **dadurch gekennzeichnet, dass** die Verdichtung mittels einer Quetschschraube oder einer Doppelwalzenpresse mit einem Verdichtungsverhältnis von 1:1 bis 8:1, vorzugsweise von 2:1 bis 5:1, vorgenommen wird.

5. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** die Pektinase enthaltende Flüssigkeit ein Enzympräparat mit pektolytischer Wirkung sowohl auf Pektine als auch veresterter Pektine enthält.

6. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** die wässrige Flüssigkeit zwei oder mehr Enzympräparate enthält, wobei mindestens eines der Präparate eine Pektinasewirkung entfaltet.

7. Verfahren nach einem der Ansprüche 1 bis 5, **dadurch gekennzeichnet, dass** die Pektinase als biologischer Wirkstoff zugesetzt wird, welcher einen oder mehrere Pilze oder Bakterien enthält, von denen einer bzw. eines eine pektolytische Wirkung besitzt.

8. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** die Pektinase aus einer Gruppe von Mikroorganismen stammt, welche *Aspergillus aculeatus* und *Aspergillus oryzae* enthält.

9. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** der Einsatz an Pektinase 2.000.000 bis 200.000.000 Polygalakturonase-Einheiten pro Tonne Fasermaterial, vorzugsweise 10.000.000 bis 50.000.000 Polygalakturonase-Einheiten/Tonne, beträgt.

10. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** die wässrige Flüssigkeit mindestens ein Mittel zur Chelatbildung enthält, vorzugsweise Diethylen-Tetramin-Pentaessigsäure in einer Einsatzmenge von 1 bis 10 kg/Tonne und/oder Sulfit in einer Einsatzmenge von 5 bis 50 kg/Tonne.

11. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** nach dem Aufnehmen der Imprägnierflüssigkeit eine Verweildauer von 3 Minuten bis 24 Stunden, vorzugsweise 15 bis 240 Minuten und besonders bevorzugter Weise von 30 bis 120 Minuten vorgesehen ist.

12. Verfahren nach Anspruch 11, **dadurch gekennzeichnet, dass** die Temperatur während der Verweildauer nach dem Aufnehmen der Imprägnierflüssigkeit 20 bis 100 °C, vorzugsweise 35 bis 70 °C und besonders bevorzugter Weise etwa 50 °C beträgt.

13. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** der pH-Wert in der Im-

prägnierflüssigkeit 3 bis 10 beträgt, vorzugsweise 4 bis 7 und besonders bevorzugter Weise etwa 5.

14. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** das Zerfasern und Mahlen des Fasermaterials unter Verwendung eines Einscheiben-, Doppelscheiben- oder konischen Refiners in einer oder mehreren Stufen vorgenommen wird.

15. Verfahren nach Anspruch 14, **dadurch gekennzeichnet, dass** die Umlaufgeschwindigkeit des Refiners 1.000 bis 3.000 UpM, vorzugsweise 1.500 bis 2.600 UpM, beträgt.

16. Verfahren nach Anspruch 14, **dadurch gekennzeichnet, dass** das Fasermaterial 2 bis 10 Minuten lang vor der Mahlung vorgewärmt wird, dass der Druck im Refiner vom atmosphärischen Druck bis zu 5 bar, vorzugsweise bis zu 4 bar, beträgt und dass die Umlaufgeschwindigkeit des Refiners vorzugsweise 1.200 bis 1.800 UpM beträgt.

17. Verfahren nach Anspruch 14, **dadurch gekennzeichnet, dass** das Fasermaterial 3 bis 30 Sekunden lang vor der Mahlung vorgewärmt wird, dass der Druck im Refiner von 4 bis 8 bar beträgt, vorzugsweise von 5 bis 8 bar, und dass die Umlaufgeschwindigkeit des Refiners vorzugsweise über 2.000 UpM liegt.

18. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** das Fasermaterial aus Nadelholzspänen oder Hartholzspänen besteht.

19. Verfahren nach einem der Ansprüche 1 bis 17, **dadurch gekennzeichnet, dass** das Fasermaterial aus anderem Fasermaterial als Holzspänen besteht, das Zuckerrohr, Bambus, Schilfrohr und Stroh umfasst.

20. Verfahren nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** der nach Zerfaserung und Mahlung erhaltene Holzstoff gebleicht wird, vorzugsweise mit einem basischen Peroxid, um so einen gebleichten Holzstoff von hoher Helligkeit zu erhalten.

21. Holzstoff. **dadurch gekennzeichnet, dass** er mit einem Verfahren nach einem der Ansprüche 1 bis 20 hergestellt ist.

Revendications

1. Procédé pour la production d'une pâte à papier mécanique, dans lequel, pour réduire la consommation d'énergie de raffinage tout en améliorant les propriétés optiques et de résistance mécanique de la pâte mécanique produite, le procédé comprend les étapes consistant à :

a) comprimer initialement une matière en fibres ;
b) affaiblir sélectivement une région enrichie en pectine dans les parois des fibres de la matière en fibres en imprégnant la matière en fibres avec un liquide aqueux contenant une pectinase, avec pour résultat l'hydrolyse des pectines ;
c) défibrer et raffiner la matière en fibres pour produire une pâte mécanique.

2. Procédé suivant la revendication 1, **caractérisé en ce que** ladite compression initiale de la matière en fibres est une compression mécanique, de préférence combinée avec un prétraitement thermique de la matière en fibres, de préférence par étuvage, avant l'imprégnation.

3. Procédé suivant la revendication 2, **caractérisé en ce que** l'étuvage est effectué, de préférence à la pression atmosphérique, pendant 1 à 30 min, de préférence 10 à 20 min.

4. Procédé suivant la revendication 2 ou 3, **caractérisé en ce que** la compression est effectuée par une vis de compression ou une presse à deux cylindres, avec un rapport de compression de 1:1 à 8:1, de préférence de 2:1 à 5:1.

5. Procédé suivant l'une quelconque des revendications précédentes, **caractérisé en ce que** le liquide contenant une pectinase comprend une préparation enzymatique ayant une activité pectolytique à la fois pour les pectines et les pectines estérifiées.

6. Procédé suivant l'une quelconque des revendications précédentes, **caractérisé en ce que** le liquide aqueux comprend deux ou plus de deux préparations enzymatiques, au moins une des préparations ayant une activité de

pectinase.

7. Procédé suivant l'une quelconque des revendications 1 à 5, **caractérisé en ce que** la pectinase est ajoutée comme agent biologique comprenant un ou plusieurs champignons ou une ou plusieurs bactéries, dont au moins l'un a une activité pectolytique.

8. Procédé suivant l'une quelconque des revendications précédentes, **caractérisé en ce que** la pectinase est issue d'un groupe de micro-organismes contenant *Aspergillus aculeatus* et *Aspergillus oryzae.*

9. Procédé suivant l'une quelconque des revendications précédentes, **caractérisé en ce que** la charge de pectinase est de 2 000 000 à 200 000 000 d'unités de polygalacturonase/ tonne de matière en fibres, de préférence de 10 000 000 à 50 000 000 d'unités de polygalacturonase/tonne.

10. Procédé suivant l'une quelconque des revendications précédentes, **caractérisé en ce que** le liquide aqueux comprend au moins un agent chélatant, de préférence l'acide diéthylène-tétraminepentaacétique en une charge de 1 à 10 kg/tonne et/ou un sulfite en une charge de 5 à 50 kg/tonne.

11. Procédé suivant l'une quelconque des revendications précédentes, **caractérisé en ce qu'**un temps de rétention, après absorption du liquide d'imprégnation, va de 3 min à 24 heures, avantageusement de 15 à 240 min, et plus avantageusement de 30 à 120 min.

12. Procédé suivant la revendication 11, **caractérisé en ce qu'**une température dans la rétention après absorption du liquide d'imprégnation est de 20 à 100°C, avantageusement de 35 à 70°C et plus avantageusement égale à environ 50°C.

13. Procédé suivant l'une quelconque des revendications précédentes, **caractérisé en ce qu'**un pH dans le liquide d'imprégnation est de 3 à 10, avantageusement de 4 à 7 et plus avantageusement égal à environ 5.

14. Procédé suivant l'une quelconque des revendications précédentes, **caractérisé en ce que** le défibrage et le raffinage de la matière de fibres sont effectués en utilisant des raffineurs à un disque, à deux disques ou coniques dans une ou plusieurs étapes.

15. Procédé suivant la revendication 14, **caractérisé en ce qu'**une vitesse de rotation du raffineur est de 1000 à 3000 tr/min, de préférence de 1500 à 2600 tr/min.

16. Procédé suivant la revendication 14, **caractérisé en ce que** la matière en fibres est préchauffée pendant 2 à 10 min avant raffinage, qu'une pression du raffineur va de la pression atmosphérique jusqu'à 5 bars, de préférence jusqu'à 4 bars, et qu'une vitesse de rotation du raffineur est de préférence de 1200 à 1800 tr/min.

17. Procédé suivant la revendication 14, **caractérisé en ce que** la matière en fibres est préchauffée pendant 3 à 30 secondes avant raffinage, qu'une pression du raffineur va de 4 à 8 bars, de préférence de 5 à 8 bars, et qu'une vitesse de rotation du raffineur est de préférence supérieure à 2000 tr/min.

18. Procédé suivant l'une quelconque des revendications précédentes, **caractérisé en ce que** ladite matière en fibres consiste en copeaux de bois résineux ou copeaux de bois de feuillus.

19. Procédé suivant l'une quelconque des revendications 1 à 17, **caractérisé en ce que** la matière en fibres est une matière en fibres, ne consistant pas en fibres de bois, comprenant la bagasse, le bambou, le roseau et la paille.

20. Procédé suivant l'une quelconque des revendications précédentes, **caractérisé en ce que** la pâte obtenue après défibrage et raffinage est blanchie, de préférence avec un peroxyde alcalin, pour obtenir une pâte blanchie ayant une grande brillance.

21. Pâte mécanique, **caractérisée en ce qu'**elle a été produite par un procédé selon l'une quelconque des revendications 1 à 20.

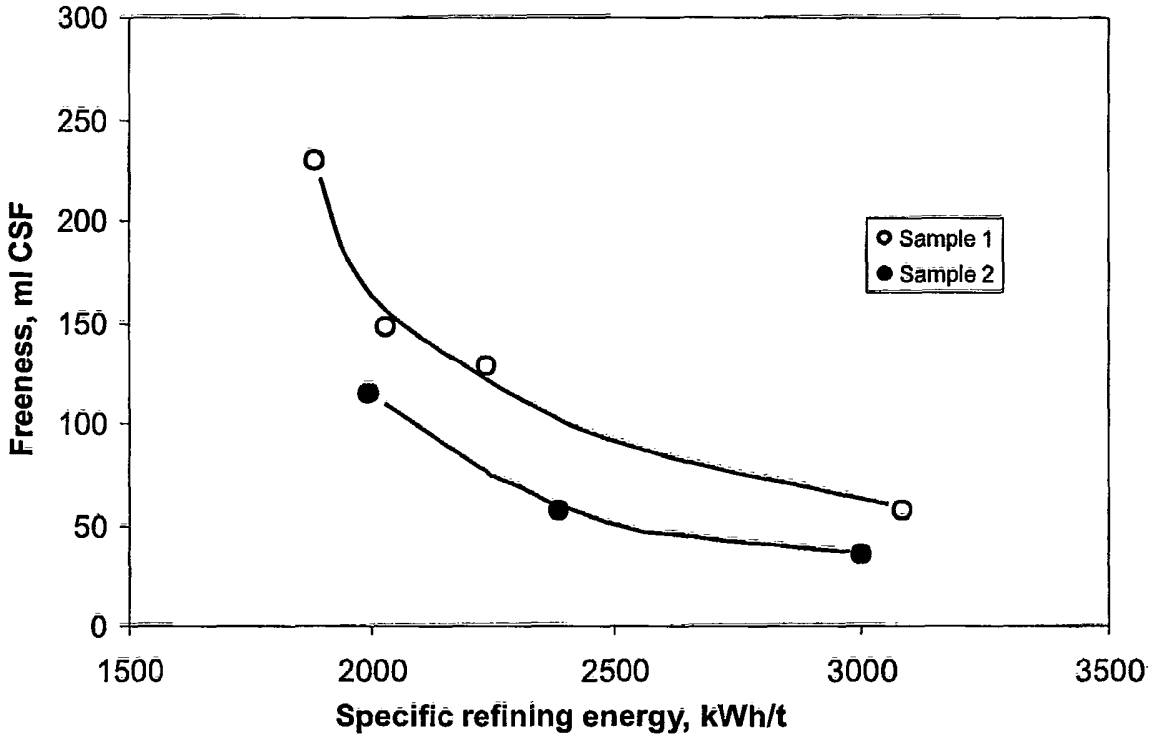
300
250
200
150
100
50
0
Freeness, ml CSF
1500
2000
2500
3000
3500
Specific refining energy, kWh/t
Sample 1
Sample 2

Fig. 1

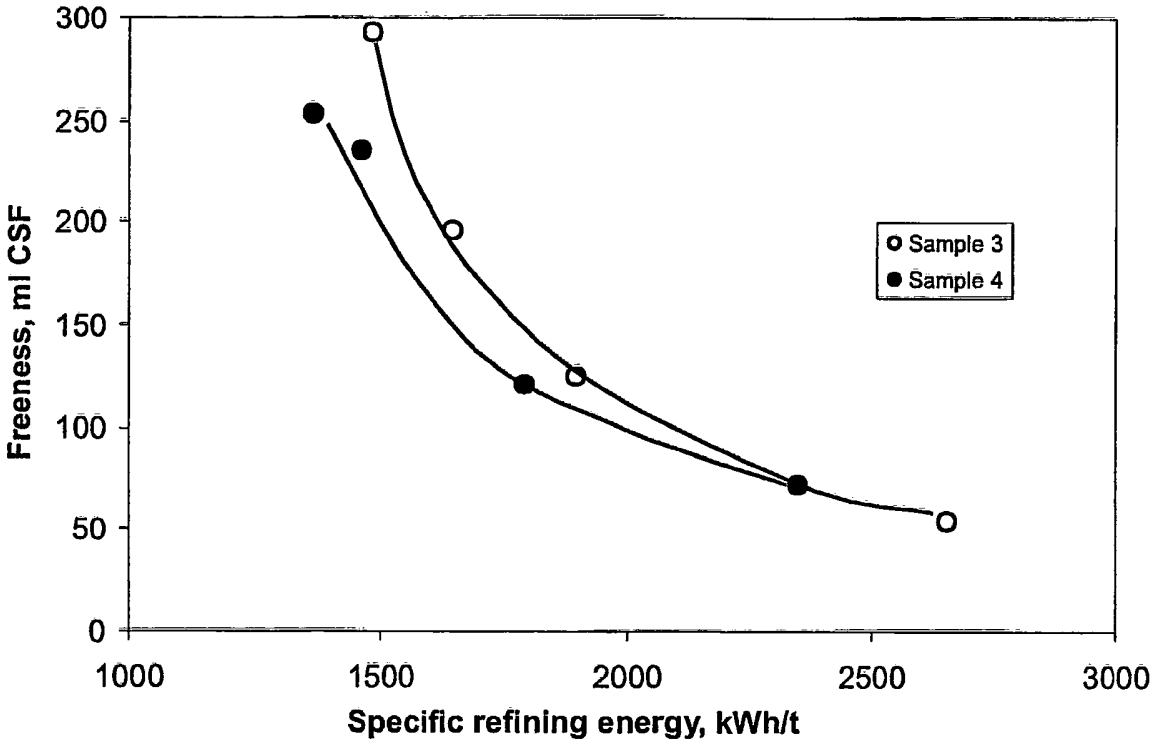
300
250
200
150
100
50
0
Freeness, ml CSF
1000
1500
2000
2500
3000
Specific refining energy, kWh/t
Sample 3
Sample 4

Fig. 2

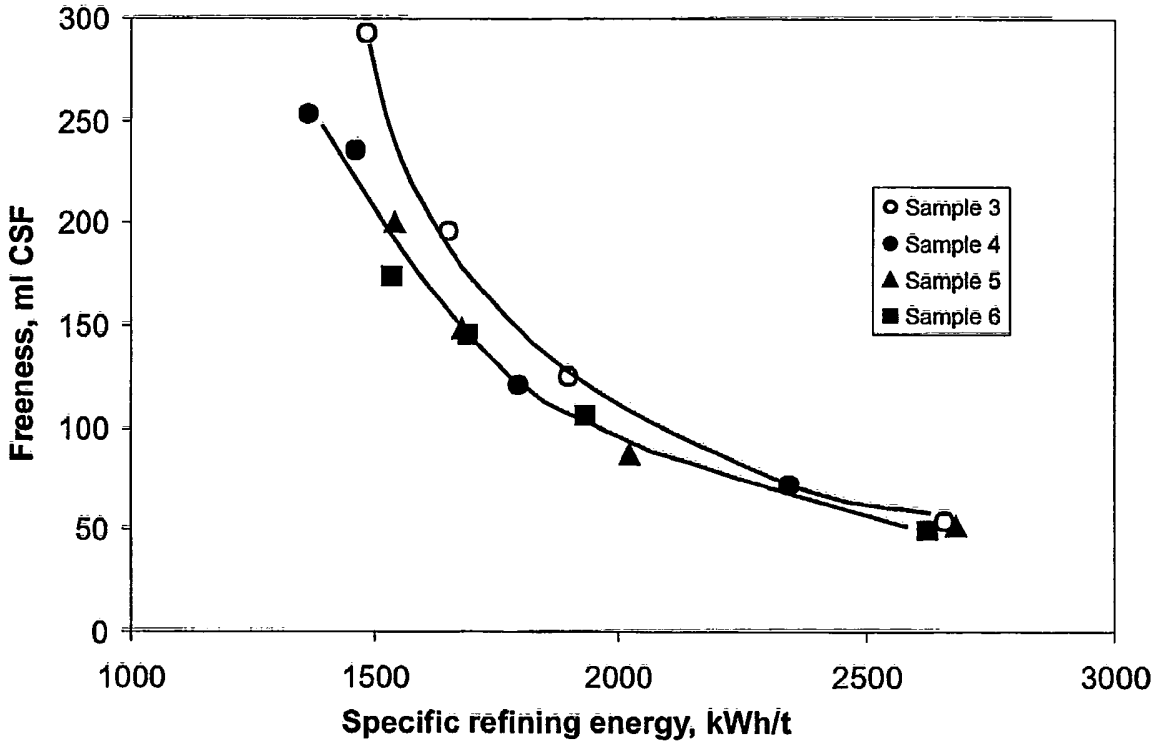
300
250
200
150
100
50
0
Freeness, ml CSF
1000
1500
2000
2500
3000
Specific refining energy, kWh/t
Sample 3
Sample 4
Sample 5
Sample 6

Fig. 3

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- WO 9740194 A **[0003]**
- US 5055159 A **[0004]**
- US 5487812 A **[0005]**
- EP 0430915 A **[0006]**
- WO 8902951 A **[0007]**

Non-patent literature cited in the description

- **HAFREN J. ; WESTERMARK U.** *Nordic Pulp and Paper,* 2001, vol. 16 (4), 284-290 **[0011]**