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(54) **METHOD FOR PRODUCING HT-CTMP HAVING HIGH TENSILE STRENGTH**

(57) There is provided a bleached high temperature chemithermomechanical pulp and a method for producing said HT-CTMP comprising the steps:

- providing wood chips comprising softwood, hardwood or mixtures thereof;
- impregnating the wood chips with an impregnation liquid to obtain impregnated chips;
- transferring the impregnated chips to a heating zone without compressing the impregnated chips;

d) heating the impregnated chips in the heating zone with steam having a temperature of above 140 °C, such as at least 150 °C, such as at least 160 °C, to obtain pre-treated chips;

- defibrating the pre-treated chips to obtain a pulp;
- bleaching the pulp to obtain a bleached pulp; and
- refining the bleached pulp at a consistency of 3-8 wt. % to obtain bleached HT-CTMP.

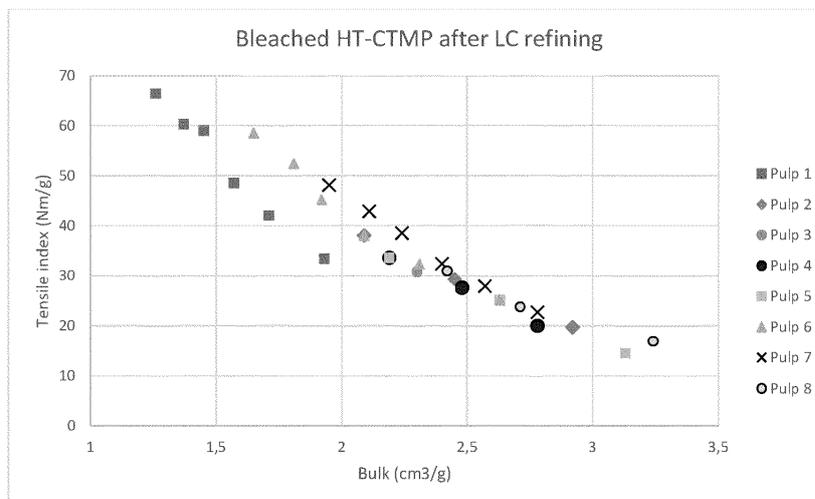


Fig. 7

Description

TECHNICAL FIELD

5 **[0001]** The present disclosure relates to the production of bleached high temperature chemithermomechanical pulp (HT-CTMP).

BACKGROUND

10 **[0002]** Chemithermomechanical pulp, CTMP, is a high yield pulp which can provide a high bulk and has been used since 1960s. CTMP is produced by mild chemical impregnation of wood chips, followed by a heat treatment to soften the wood. The treated wood chips are then subjected to defibration/refining and optionally bleaching. The obtained CTMP typically has comparatively high bulk, preferably in combination with low shives content. The process can be further improved by using higher temperatures during the heat treatment. Using temperatures of at least 140 °C during
15 the heat treatment leads to a decrease in the energy input needed during the pressurized defibration step. However, CTMP and especially high temperature CTMP (HT-CTMP) has a poor fiber bonding ability and hence low tensile strength, limiting its use.

SUMMARY

20 **[0003]** The present inventors have realized that a bleached HT-CTMP having particularly good fiber bonding ability and thereby a high tensile strength can be produced if the process comprises low-consistency refining subsequent to the bleaching of the pulp.

25 **[0004]** According to a first aspect of the present disclosure, there is provided a method for producing bleached HT-CTMP comprising the steps:

- a) providing wood chips comprising softwood, hardwood or a mixture thereof;
- b) impregnating the wood chips with an impregnation liquid to obtain impregnated chips;
- 30 c) transferring the impregnated chips to a heating zone without compressing the impregnated chips;
- d) heating the impregnated chips in the heating zone with steam having a temperature of above 140 °C, such as at least 150 °C, such as at least 160 °C, to obtain pre-treated chips;
- 35 e) defibrating the pre-treated chips to obtain a pulp;
- f) bleaching the pulp to obtain a bleached pulp; and
- 40 g) refining the bleached pulp at a consistency of 3-8 wt. %.

[0005] By producing a bleached HT-CTMP according to the first aspect, a bleached HT-CTMP having high tensile strength may be obtained. Positioning the bleaching step prior to the low consistency refining of step g) surprisingly enables an improved effect of the low consistency refining and hence a development of the strength of the produced
45 HT-CTMP that is not possible otherwise.

[0006] The bleaching in step f) may be a two-step process. In the two-step bleaching process, the first and the second bleaching step may be performed at different consistencies such as the first step occurring at a medium consistency of 10-12 wt.% and the second step occurring at a high consistency of 30-35 wt. %. The two-step bleaching processes facilitates the bleaching of the pulp to a high brightness.

50 **[0007]** The defibration of the treated chips in step e) may occur at a solid content of 40-50 wt.%. By having a solids content of 40-50 wt.% a more effective defibration can be obtained in step e).

[0008] Furthermore, the pressure during defibration in step e) may be 6-12 bar. The pressure may be lower at the beginning of the defibration and then increase throughout the defibration step. The high pressure during step e) may facilitate the defibration.

55 **[0009]** The low consistency (LC) refining in step g) may be performed using multiple parallel LC refiners.

[0010] The consistency in step g) is preferably 3-7 wt.%, such as 4-6 wt.%. An advantage to using low consistencies during step g) is that the second refining step may than be less expensive.

[0011] The method may further comprise dewatering of the pulp prior to bleaching of step f). The dewatering may

improve the bleaching procedure. Due to the energy-efficiency of the refining of step g), the freeness prior to bleaching may be kept relatively high (by a relatively low degree of defibration in step e)), which means that the pulp is relatively easily dewatered. In other words, the method of the first aspect enables energy efficient dewatering prior to the bleaching of step f).

5 **[0012]** The pulp obtained in step e) may have a Canadian Standard Freeness (CSF) of 650-750 ml, such as 690-750 ml as measured according to ISO 5267-2:2001 after disintegration according to ISO 5263-3:2004. As explained above, such a high freeness enables easier dewatering in the following washing steps which in turn can reduce the required energy input and/or improve the dewatering.

10 **[0013]** The impregnation liquid, which is aqueous, may comprise NaOH and/or Na₂SO₃. The temperature of the impregnation liquid is preferably at least 70°C, such as 70°C-99°C, such as 80°C-99°C. At such a relatively high temperature, the viscosity of the impregnation liquid is lower, which facilitates the absorption thereof.

[0014] The method may further comprise pre-steaming of the wood chips prior to the impregnation in step b). The pre-steaming may remove air present within the chips and condition the chips, thereby improving the following impregnation.

15 **[0015]** The wood chips may further be feed to the impregnation liquid via a plug screw wherein the wood chips are compressed. The compression may enable quick expansion once the wood chips come in contact with the impregnation liquid and hence improve the impregnation.

[0016] The residence time in step b) is preferably no more than two minutes.

20 **[0017]** According to a second aspect, a bleached HT-CTMP is provided. The HT-CTMP has a tensile index measured according to ISO 1924-3:2005 after sheet forming according to ISO 5269-1:2005 of at least 45 Nm/g, such as at least 50 Nm/g, such as at least 55 Nm/g. An upper limit for the tensile index may be 70 Nm/g. For higher bulk values, the upper limit may be 60 Nm/g.

[0018] Furthermore, the bleached HT-CTMP may have a bulk of at least 1.25 cm³/g, such as at least 1.35 cm³/g, such as at least 1.60 cm³/g. An upper limit for the bulk may be 2.00 cm³/g. For higher tensile index values, the upper limit may be 1.70 cm³/g. Bulk is measured according to ISO 534:2011 after sheet forming according to ISO 5269-1:2005.

25 **[0019]** As shown in figure 7 below, four different HT-CTMPs having a bulk in the range of 1.60-2.00 cm³/g and a tensile index in the range of 45-60 Nm/g have been prepared. Further, four different HT-CTMPs having a bulk in the range of 1.25-1.70 cm³/g and a tensile index in the range of 55-70 Nm/g have been prepared.

[0020] The CSF of the bleached HT-CTMP may be 200-460 ml as measured according to ISO 5267-2:2001 after disintegration according to ISO 5263-3:2004.

30 **[0021]** The bleached HT-CTMP may comprise at least 95 dry wt.%, such as at least 100 dry wt.%, hardwood fibres, such as birch fibres.

[0022] High temperature chemithermomechanical pulp, HT-CTMP, is defined as CTMP produced according to a process in which impregnated chips are heated with steam having a temperature of above 140 °C, such as at least 150 °C, such as at least 160 °C. An upper limit may be 190 °C.

35 BRIEF DESCRIPTION OF THE DRAWINGS

[0023] Aspects and embodiments are now described, by way of example, with reference to the accompanying drawings, in which:

40 Fig 1 schematically illustrates a system for producing bleached HT-CTMP.

Fig 2 schematically illustrates a continuation of the system in Fig 1 designed for producing market HT-CTMP.

45 Fig 3 schematically illustrates a continuation of the system in Fig 1 designed to produce market HT-CTMP and supply an adjacent board-making machine with HT-CTMP.

50 Fig 4a shows the bulk as a function of refining energy input for bleached pulp 4 and non-bleached pulp 4*. The series starting with the filled triangle represents the unbleached pulp and the series starting with the unfilled triangle represents the bleached pulp.

Fig 4b shows the bulk as a function of refining energy input for bleached pulp 6 and non-bleached 6*. The series starting with the filled diamond represents the unbleached pulp and the series starting with the unfilled diamond represents the bleached pulp.

55 Fig 5a shows the tensile index as a function of refining energy input for bleached pulp 4 and non-bleached pulp 4*. The series starting with the filled triangle represents the unbleached pulp and the series starting with the unfilled triangle represents the bleached pulp.

Fig 5b shows the tensile index as a function of refining energy input for bleached pulp 6 and non-bleached 6*. The series starting with the filled diamond represents the unbleached pulp and the series starting with the unfilled diamond represents the bleached pulp.

5 Fig 6a shows the relationship between bulk and tensile index for bleached pulp 4 and non-bleached pulp 4*. The series starting with the filled triangle represents the unbleached pulp and the series starting with the unfilled triangle represents the bleached pulp.

10 Fig 6b shows the relationship between bulk and tensile index for bleached pulp 6 and non-bleached 6*. The series starting with the filled diamond represents the unbleached pulp and the series starting with the unfilled diamond represents the bleached pulp.

Fig 7 shows the relationship between bulk and tensile index of the tested bleached pulps.

15 Fig 8 shows the relationship between tensile index and CSF of the tested bleached pulps.

DETAILED DESCRIPTION

20 **[0024]** The present disclosure relates to a production method of a HT-CTMP having improved fiber bonding ability and thereby improved properties such as tensile strength. The HT-CTMP of the present disclosure overcomes the shortcomings associated with HT-CTMP and opens up a variety of applications in which CTMP or HT-CTMP previously could not be used. In some embodiments, the properties of the obtained HT-CTMP may even be similar to Kraft pulp. A HT-CTMP of similar properties as Kraft pulp is advantageous since HT-CTMP is produced in a much higher yield than Kraft pulp.

25 **[0025]** A HT-CTMP process typically gives rise to a high yield pulp having high bulk and relatively low tensile strength. The low tensile strength is due to poor fiber bonding. The fibers in HT-CTMP have a smooth surface as they are "coated" with lignin, which gives the poor fiber bonding ability. The smooth fibers are difficult to refine and require a high amount of refining energy to develop a relatively low degree of tensile strength. The present inventors have realized that by bleaching the pulp prior to a second refining step at low consistency, a more efficient refining may be obtained and hence
30 a higher tensile strength may be developed at a lower refining energy consumption.

[0026] Not wishing to be bound by any theory, it is believed that the bleaching reacts with the lignin coated on the fibers and increases the surface roughness of the fibers enabling a better refining. The energy requirement for refining the pulp to a certain freeness may be three times smaller for the bleached pulp compared to the non-bleached pulp.

35 **[0027]** Hardwood such as birch, maple and aspen as well as softwood such as spruce and pine may be used to produce the HT-CTMP of the present disclosure. It is further possible to modify the final properties of the HT-CTMP by using mixtures of hardwood and softwood e.g. to obtain an optimal relationship between long and short fibers.

[0028] Figures 1-3 illustrate exemplary embodiments of a system for producing bleached HT-CTMP.

40 **[0029]** A chipper 101 maybe used to prepare chips from wood. In case of hardwood, in particular maple, it is preferable to prepare chips that are relatively short, such as < 20 mm, to aid impregnation. Softwood chips are generally easier to impregnate and can hence be longer, such as 22-24 mm. The settings of a conventional wood chipper can be adjusted to achieve such lengths. The thickness of the chips is proportional to its length, i.e. the shorter the chips are, the thinner they will be.

45 **[0030]** The chips from the chipper 101 are typically stored in at least one silo 102. When different types of wood are mixed in the system, there is typically one silo for each type of wood, such as one hardwood chips silo 102a and one softwood chips silo 102b. When there is more than one type of wood chips, a chips mixing system 103 is preferably arranged downstream of the silos 102a, 102b.

50 **[0031]** The chips from the chips mixing system 103 are optionally stored in a maturation silo 104 for a period of at least 24 h (typically about 72 h). A typical temperature in the maturation silo 104 is 60°C, which can be achieved by feeding low-pressure steam into the maturation silo 104. The treatment of the chips in the maturation silo 104 degrades triglycerides. The degradation products can then be more easily extracted in downstream process steps.

[0032] Another option is to design the chip silos 102a, 102b as maturation silos. A benefit of this option is that the maturation time and temperature can be individually adapted to the respective wood types.

[0033] Yet another option is to place the maturation silo 104 between the chip washing arrangement 106 and the pre-steaming bin 107 described below. It is also possible to omit the maturation step.

55 **[0034]** Before being impregnated, the chips may be washed in a chip washing arrangement 106. Upstream the chip washing arrangement 106, a conditioning device 105 may be arranged. The conditioning device 105 is typically a chip steaming bin. The purpose of the conditioning device 105 is to provide chips of fairly constant temperature. The conditioning device 105 may also, to some extent, reduce variations in the moisture content of the chips. During cold winter

months, the conditioning device 105 may be used to melt the ice on the chips, which facilitates the downstream washing and processing. Hence, the conditioning device 105 may be particularly advantageous when there is no upstream maturation silo.

5 **[0035]** In the chip washing arrangement 106, the chips are typically soaked and agitated in water and then dewatered. Dirt and gravel are removed in the chip washing arrangement 106. The washed and dewatered chips are then steamed in a pre-steaming bin 107. The residence time of the chips in the pre-steaming bin 107 is typically at least 10 min such as 15-20 min. The pre-steaming of the chips removes air from the chips and replaces it with steam leading to an improved impregnation downstream.

[0036] The steamed chips from the pre-steaming bin 107 are subjected to impregnation in one or two steps.

10 **[0037]** In case of one-step impregnation, a plug screw 108 may feed the steamed chips into a reactor 109. The steamed chips, which were compressed in the plug screw 108, expand in a bath of aqueous impregnation liquid 110 in the reactor 109. During the expansion, the chips absorb impregnation liquid. The temperature of the impregnation liquid is preferably 80°C-99°C. The expanded and impregnated chips are lifted from the bath of impregnation liquid 110 by means of a transport screw 111 and are then allowed to fall over an edge 112 and into a heating zone 113 of the reactor 109, in which they are heated by steam having a temperature above 140°C. The transferring of the impregnated chips from the bath of impregnation liquid 110 to the heating zone 113 occurs without any compression of the chips. The impregnation liquid is thereby not pressed out of the chips prior to reaching the heating zone 113. The chips treated in the reactor 109 are transferred to a chips defibrator 114 without flashing off any steam on the way.

15 **[0038]** In case of two-step impregnation, a plug screw 115 may feed the steamed chips into a pre-impregnation chamber 116. The steamed chips, which were compressed in the plug screw 115, expands in a bath of pre-impregnation liquid 117 in the pre-impregnation chamber 116. During the expansion, the chips absorb pre-impregnation liquid. The temperature of the pre-impregnation liquid is preferably 80°C-99°C. The pre-impregnation liquid is water that may comprise NaOH and optionally Na₂SO₃. The expanded and impregnated chips are lifted from the bath of pre-impregnation liquid 117 by means of a transport screw 118. A plug screw 119 may then feed the pre-impregnated chips into a reactor 120. 20 The pre-impregnated chips, which were compressed in the plug screw 119, expand in a bath of impregnation liquid 121 in the reactor 120. During the expansion, the chips absorb impregnation liquid, which preferably has a temperature of 80°C-99°C. The expanded and impregnated chips are lifted from the bath of impregnation liquid 121 by means of a transport screw 122 and are then allowed to fall over an edge 123 and into a heating zone 124 of the reactor 120, in which they are heated by steam having a temperature above 140°C. The transferring of the impregnated chips from the bath of impregnation liquid 121 to the heating zone 124 occurs without any compression of the chips. The impregnation liquid is thereby not pressed out of the chips prior to reaching the heating zone 124. The chips treated in the reactor 120 are transferred to the chips defibrator 114 without flashing off any steam on the way.

25 **[0039]** The impregnation liquid, in both the one-step and the two-step impregnation, may comprise NaOH, Na₂SO₃ or mixtures thereof. The NaOH softens the wood chips by modification of the carbohydrates such as hemicellulose and is typically used more for hardwood. The Na₂SO₃ softens the wood by modifying the lignin by sulphonation making it more hydrophilic and is typically used more for softwood. The impregnation liquid may be adjusted as to optimize the softening of the chips which may occur differently depending on the wood composition.

30 **[0040]** In the chips defibrator 114, the dry matter content is 40-50 wt. % such as 45-50 wt.% (in case there is no plug screw between the heating zone 124 and the chips defibrator 114, the dry matter content may however be as low as 30%). It is advantageous for the solid content to be high in order to use as much of the refining energy for defibrating rather than vaporizing the water in the impregnated chips. However, too high solid content, >50 wt.%, may lead to burnt fibers. The pressure at the beginning of the defibration step is typically 6-7 bar and may increase to 10-12 bar during the duration of the defibration step. The defibrated chips from the chips defibrator 114 may be subjected to flashing in a steam separator 125 and then pulped in a first pulper 126. The pulp from the first pulper 126 may then be treated in 45 a first dewatering press 127. The pressate from the first dewatering press 127 contains extractives (and dissolved wood substances and residual chemicals) that are unwanted in the final HT-CTMP product. Separation of extractives by pressing in this position is advantageous since the pulp still has very high freeness (typically >650 ml or even >700 ml) and is thus easily dewatered. Limiting the residence time in the first pulper 126 to below 10 min (typically about 3 min) is advantageous since it limits the time available for the extractives to be re-adsorbed onto the fibers before the first dewatering press 127.

50 **[0041]** The pulp from the first dewatering press 127 is subjected to middle consistency (MC) bleaching in a MC bleach tower 128 using unreacted peroxide from a downstream high consistency (HC) bleaching. Fresh alkali such as NaOH may be added to the MC bleaching if necessary. MC means 10-12 wt.%. The MC-bleached pulp may be dewatered in a second dewatering press 129 also producing a pressate. The pulp from the second dewatering press is subjected to high consistency (HC) bleaching in a HC bleach tower 130 using fresh peroxide and alkali. It is advantageous that the hydrogen peroxide is present in excess compared to the alkali during the entire bleaching process in order to obtain a high brightness. The HC-bleached fibers from the HC bleach tower 130 may be pulped in a second pulper 131 (residence time is preferably <10 min, such as about 3 min) to produce a pulp having a consistency of about 3-8 wt.%. This pulp 55

is then subjected to low consistency (LC) refining in LC refiners 132. The LC refining may be performed in multiple stages, typically three LC refiners are used. A third dewatering press 133 may then separate a third pressate from the LC-refined pulp. The fibers from the third dewatering press 133 may be pulped in a third pulper 134 (residence time is preferably <10 min, such as about 3 min) to produce a pulp having a consistency of 2-4 wt.%. Screens 135 may then be used to separate a reject from the pulp from the third pulper 134. The separated reject is collected in a reject tank 136.

[0042] The design of the remaining parts of the system depends on if only market pulp is produced (i.e. all HT-CTMP is subjected to flash drying and baling) or if there is also an adjacent board-making machine to which at least part of the HT-CTMP is supplied without drying.

[0043] In the former case, which is illustrated in figure 2, the pulp from the screens 135 are cleaned in cyclones 137 to provide cleaned pulp and a second reject that is collected in a second reject tank 138. The cleaned pulp is then filtered in a disc filter 139 and collected in a MC tower 140. From the pulp from the MC tower 140, a fourth dewatering press 141 produces dewatered fibers and a fourth pressate. The dewatered fibers are led to an arrangement for fiber treatment and shredding 142 and then to a flash drying arrangement 143. Finally, bales of the dried fibers from the flash drying arrangement 143 are formed in a baling arrangement 144.

[0044] In the latter case, which is illustrated in figure 3, the pulp from the screens 135 is filtered in a disc filter 145 and treated in a fourth dewatering press 146 such that a fourth pressate and an MC pulp are obtained. The MC pulp is collected in a MC tower 147.

[0045] To produce (dried) market pulp, a fifth dewatering press 148 produces dewatered fibers and a fifth pressate from the MC pulp from the MC tower 147. The dewatered fibers are led to an arrangement for fiber treatment and shredding 149 and then to a flash drying arrangement 150. Finally, bales of the dried fibers from the flash drying arrangement 150 are formed in a baling arrangement 151.

[0046] To use the produced HT-CTMP directly in the production of paperboard, MC pulp from the MC tower 147 is led to a board-making machine.

[0047] A bleached HT-CTMP having a tensile index of at least 45 Nm/g, such as at least 50 Nm/g, such as at least 55 Nm/g can be obtained according to the present disclosure. An upper limit for the tensile index may be 70 Nm/g. For higher bulk values, the upper limit may be 60 Nm/g. The tensile index is measured according to ISO 1924-3:2005 after sheet forming according to ISO 5269-1:2005.

[0048] The bleached HT-CTMP may further have a bulk of at least 1.25 cm³/g, such as 1.35 cm³/g, such as at least 1.60 cm³/g. An upper limit for the bulk may be 2.00 cm³/g. For higher tensile index values, the upper limit may be 1.70 cm³/g. The bulk is measured according to ISO 534:2011 after sheet forming according to ISO 5269-1:2005.

[0049] By adapting the impregnation chemistry, wood composition and performing the LC refining after the bleaching; HT-CTMPs having a bulk in the range of 1.60-2.00 g/cm³ and a tensile index in the range of 45-60 Nm/g can be obtained. Another example of the present disclosure, is HT-CTMPs having a bulk in the range of 1.25-1.70 g/cm³ and a tensile index in the range of 55-70 Nm/g.

The bleached HT-CTMP may have a CSF in the range of 200-460 ml as measured according to ISO 5267-2:2001 after disintegration according to ISO 5263-3:2004.

[0050] Performing the LC refining after the bleaching enables a more effective LC refining step and hence a wider range of properties which can be obtained for the HT-CTMP.

EXAMPLES

Example 1

[0051] Pilot tests were performed using birch and spruce as raw materials in dry weight ratios of 100/0, 70/30 and 55/45. The raw materials were treated in a chipper to obtain chips which were thereafter screened using a vibrating screen equipped with hole-plates having a diameter of 35 mm and 8 mm. The screening was mainly performed in order to remove oversized chips and sawdust. The birch and spruce chips were then mixed together in different ratios.

[0052] The mixed chips were heated for about 15 min in an atmospheric pre-steaming bin to a temperature of at least 90 °C using steam. After the pre-steaming, the chips were transferred to a reactor via a plug screw. The chips were compressed in the plug screw which had a compression rate of 1:3.

[0053] The reactor comprised an impregnator (bath of impregnation liquid) and a heating zone, wherein a vertical twin-screw impregnator was used. The pre-steamed chips and the impregnation liquid were continuously feed to the impregnator at separate inlets. The impregnation liquid was feed into the impregnator at a flow rate corresponding to 0.5 m³/tonne fiber. NaOH and Na₂SO₃ were used as the impregnation chemicals at different concentrations, see table 1. In addition to these chemicals, diethylenetriamine penta acetic acid (DTPA) was added as a complexing agent in an amount of 2 kg/tonne fiber. The impregnation occurred instantaneously as the compressed chips expanded in the impregnation liquid. The impregnation liquid had a temperature of ~40°C and the liquid uptake was 2.2 m³/tonne fiber.

[0054] The impregnated chips were transferred to the heating zone of the reactor via the twin-screw. The impregnated

chips were heating at a saturated steam pressure corresponding to a temperature of 140 °C or 165 °C for 1-2 min. The pre-treated chips were then transferred to defibration. The chips were defibrated using a 20-inch OVP 20 flat disk defibrator/refiner supplied with standard 5811 Valmet pilot refiner plates. The refiner was run at 1500 rpm.

[0055] The defibrated pulp was dewatered and then bleached in a laboratory procedure. NaOH and hydrogen peroxide (H₂O₂) were used as bleaching chemicals at different ratios, see Table 1. After the bleaching, the bleached pulp was refined at a consistency of 4-6 wt.% in a laboratory LC refiner at different refining energies.

Table 1. The recipes for the different HT-CTMP produced in the pilot test.

Pulp	Chip composition	NaOH/NaSO ₃ (kg/kg per tonne)	Heat pretreatment (°C)	Bleaching H ₂ O ₂ /NaOH (kg/kg per tonne)	CSF prior to bleaching (ml)
1	100 % birch	9/9	165	60/59	547
2	100 % birch	9/9	165	60/48	547
3	100 % birch	20/20	140	60/48	495
4	100 % birch	20/20	165	60/48	631
4*	100 % birch	20/20	165	-	631
5	70% birch/30 % spruce	12/12	165	60/48	690
6	70 % birch/30 % spruce	12/12	165	60/43	690
6*	70% birch/30 % spruce	12/12	165	-	690
7	55% birch/45 % spruce	10/10	165	60/45	669
8	55% birch/45 % spruce	10/10	165	60/48	669

[0056] The tensile index was determined according to ISO 1924-3:2005 after sheet formation according to ISO 5269-1:2005. The bulk was determined according to ISO 534:2011 after sheet formatting according to ISO 5269-1:2005 and the CSF was determined according to ISO 5267-2:2001 after disintegration according to ISO 5263-3:2004.

[0057] Samples of pulp 4 and 6 were taken out prior to bleaching (designated pulp 4* and pulp 6*) to compare the effect of LC refining on bleached versus unbleached HT-CTMP. Figures 4-5 show the tensile index and the bulk of pulps 4, 4*, 6 and 6* as a function of the refining energy.

[0058] An increase in tensile index and a decrease in bulk, at the same refining energy input, was observed for bleached pulp 4 and 6 compared to unbleached pulp 4* and 6* respectively, see figures 4a-4b and 5a-5b. Furthermore, for both the bleached and unbleached pulps, a reduction in bulk and an increase in tensile index could be observed with an increase in refining energy input which was to be expected.

[0059] Surprisingly, figures 5a-5b also show a steeper increase in tensile index with increasing refining energy input for the bleached pulps (pulp 4 and 6) compared to the unbleached pulps (pulp 4* and 6*) (as can be observed by the steeper incline of the plotted values for the bleached pulps 4 and 6). This indicates that a higher tensile index can be obtained at a lower refining energy input. Hence, the LC refining is much more efficient on bleached pulp compared to unbleached pulp. The steeper increase further indicates that higher tensile indexes can be obtained by bleaching prior to LC refining.

[0060] The influence of refining energy input on bulk was similar for pulp 6* (unbleached) and pulp 6 (bleached), see figure 4a. The influence of the refining energy input was slightly larger for pulp 4 compared to pulp 4* showing a slightly steeper decrease in bulk with increased refining energy input for the bleached pulp (pulp 4) compared to the unbleached (pulp 4*), see figure 4b.

[0061] The relationship between bulk and tensile index as a function of refining energy input is different for the bleached (pulp 4 and 6) and unbleached pulps (pulp 4* and 6*), see figures 6a and 6b. When LC refining after bleaching, the bulk/tensile index curve shifts to lower bulk and high tensile index compared to LC refining prior to bleaching. Interestingly, the bulk does not decrease as much with increased tensile index for the bleached pulps (pulp 4 and 6) compared to the unbleached pulps (pulp 4* and 6*) (as can be observed by a flatter bulk/tensile index curve for the bleached pulps

compared to the unbleached pulps). This trend can be observed both for pulp 4 and pulp 6.

[0062] Figure 7 shows the relationship between bulk and tensile index, bulk/tensile index curve, at different refining energy inputs for the tested bleached pulps (the pulps were refined after bleaching), see table 1. All the tested pulps showed the same trend, a decrease in bulk and an increase in tensile index with increased refining energy input. The bulk/tensile curve was affected by the wood composition, impregnation and bleaching chemistry as can be observed in figure 7.

[0063] Figure 7, hence, shows that a HT-CTMP with a wide variety of ratios between tensile index and bulk can be obtained by varying the raw materials, impregnation chemistry and bleaching chemistry. As an example, pulp 1 comprising 100 % birch had a tensile index of 66.4 Nm/g and a bulk of 1.26 cm³/g at a specific refining energy input. Another example is pulp 6 which had a tensile index of 45.2 Nm/g and a bulk of 1.92 cm³/g at a specific refining energy input.

[0064] Figure 8 shows the relationship between tensile index and CSF at different refining energy inputs for the tested bleached pulps. The tensile index of the tested pulps increases with decreased CSF (increased refining energy input). For example, pulp 6 may, at a certain refining energy input, have a tensile index of 58.5 Nm/g and a CSF of 277 ml. At a lower refining energy input, pulp 6 may have a tensile index of 45.2 Nm/g and a CSF of 460 ml.

[0065] The results obtained in the pilot test show that by performing the bleaching prior to LC refining, a more effective LC refining is obtained. Furthermore, a more effective LC refining enables the production HT-CTMPs having versatile properties as disclosed above.

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Claims

1. A method for producing bleached high temperature chemithermomechanical pulp, HT-CTMP, comprising the steps:
 - a) providing wood chips comprising softwood, hardwood or a mixture thereof;
 - b) impregnating the wood chips with an impregnation liquid to obtain impregnated chips;
 - c) transferring the impregnated chips to a heating zone without compressing the impregnated chips;
 - d) heating the impregnated chips in the heating zone with steam having a temperature of above 140 °C, such as at least 150 °C, such as at least 160 °C, to obtain pre-treated chips;
 - e) defibrating the pre-treated chips to obtain a pulp;
 - f) bleaching the pulp to obtain a bleached pulp; and
 - g) refining the bleached pulp at a consistency of 3-8 wt. %.
2. The method of claim 1, wherein the bleaching is a two-step process.
3. The method of claim 2, wherein the bleaching in the first step occurs at a medium consistency of 10-12 wt.% and the second step occurs at a high consistency of 30-35 wt. %.
4. The method of anyone of the preceding claims, wherein the defibration of the treated chips in step e) occurs at a solid content of 40-50 wt.%.
5. The method of anyone of the preceding claims, wherein the pressure during the defibration in step e) is 6-12 bar.
6. The method of anyone of the preceding claims, wherein the pulp obtained in step e) has a Canadian Standard Freeness of 650-750 ml, such as 690-750 ml as measured according to ISO 5267-2:2001 after disintegration according to ISO 5263-3:2004.
7. The method of anyone of the preceding claims, wherein the impregnation liquid comprises NaOH and/or Na₂SO₃.
8. The method of anyone of the preceding claims, further comprising feeding the wood chips to the impregnation liquid using a plug screw.
9. The method of anyone of the preceding claims, further comprising pre-steaming of the wood chips prior to impregnation in step b).
10. A bleached high temperature chemithermomechanical pulp, HT-CTMP, having a tensile index measured according to ISO 1924-3:2005 after sheet forming according to ISO 5269-1:2005 of at least 45 Nm/g, such as at least 50 Nm/g, such as at least 55 Nm/g.

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11. The bleached HT-CTMP of claim 10, having a bulk of at least 1.25 cm³/g, such as 1.35 cm³/g, such as at least 1.60 cm³/g as measured according to ISO 534:2011 after sheet forming according to ISO 5269-1:2005.

12. The bleached HT-CTMP of claim 11 having:

a bulk in the range of 1.60-2.00 cm³/g and a tensile index in the range of 45-60 Nm/g; or
a bulk in the range of 1.25-1.70 cm³/g and a tensile index in the range of 55-70 Nm/g.

13. The bleached HT-CTMP of any one of claims 10-12, having a Canadian Standard Freeness in the range of 200-460 ml as measured according to ISO 5267-2:2001 after disintegration according to ISO 5263-3:2004.

14. The bleached HT-CTMP of any one of claims 10-13, comprising at least 95 dry wt. % hardwood fibres, such as birch fibres.

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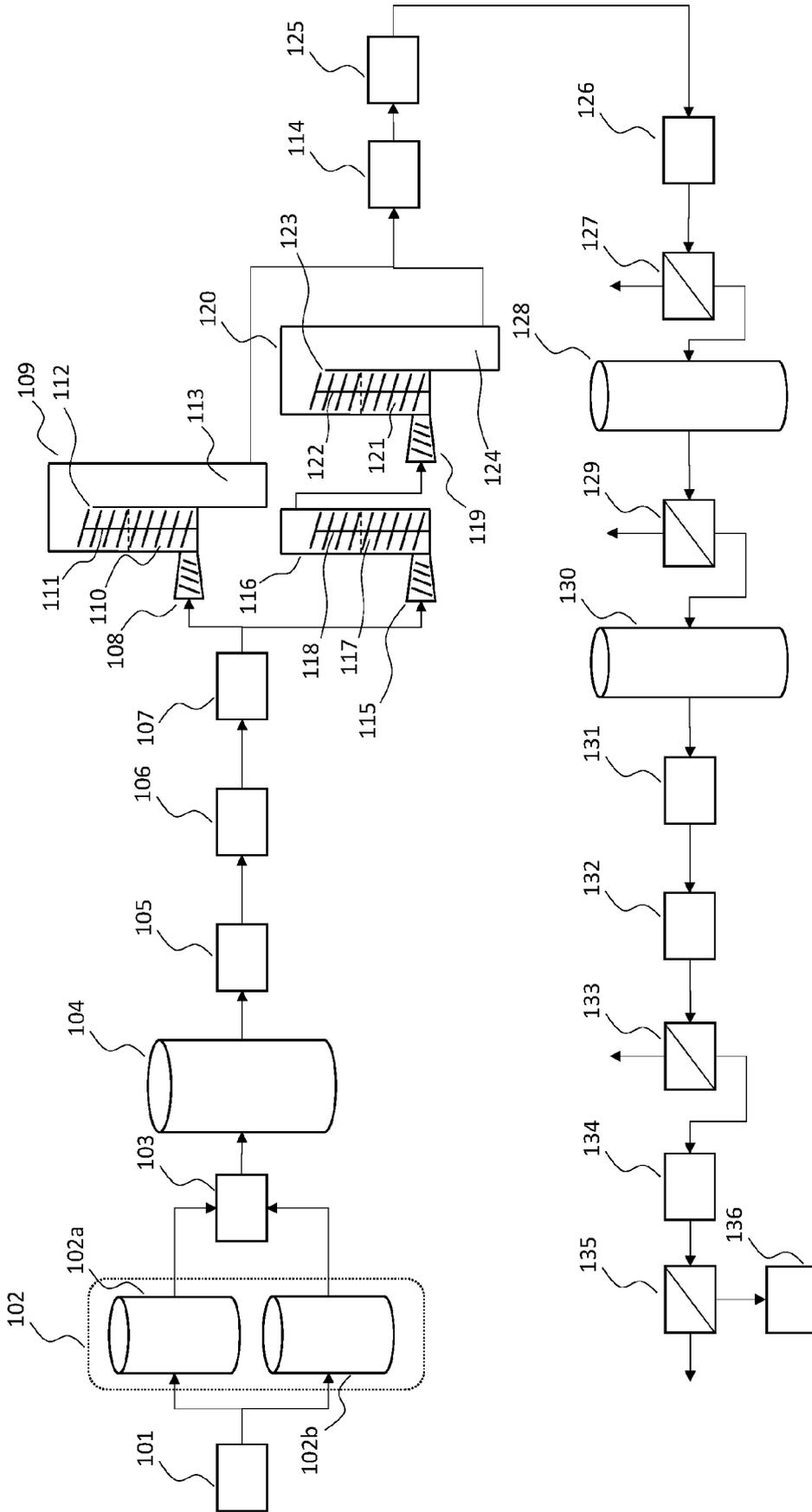


Fig. 1

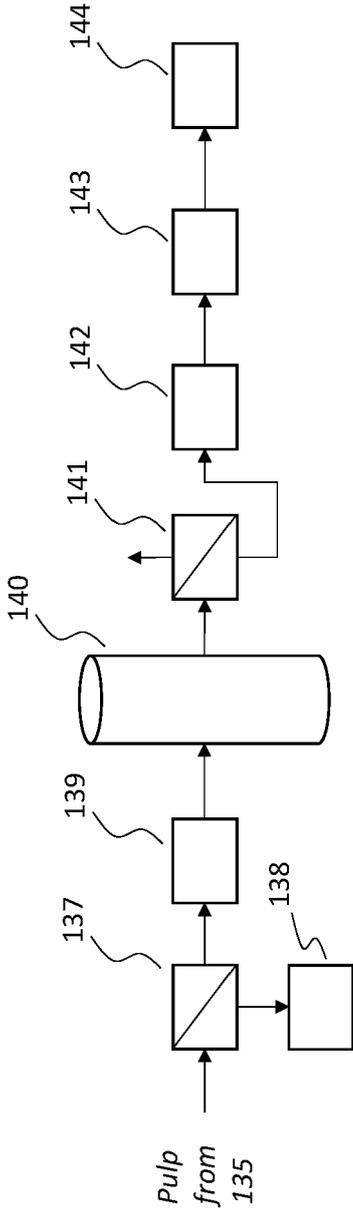


Fig. 2

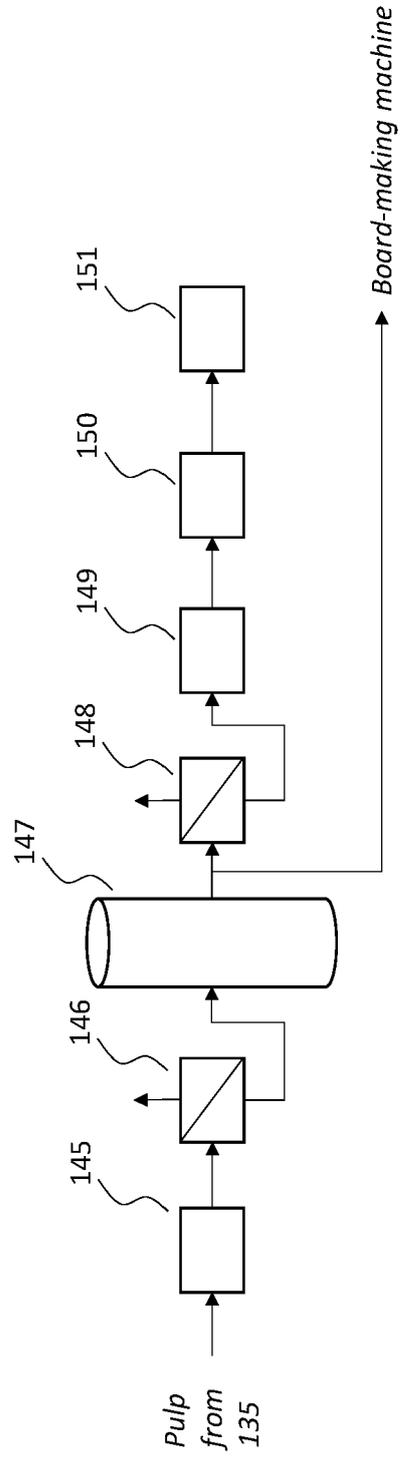


Fig. 3

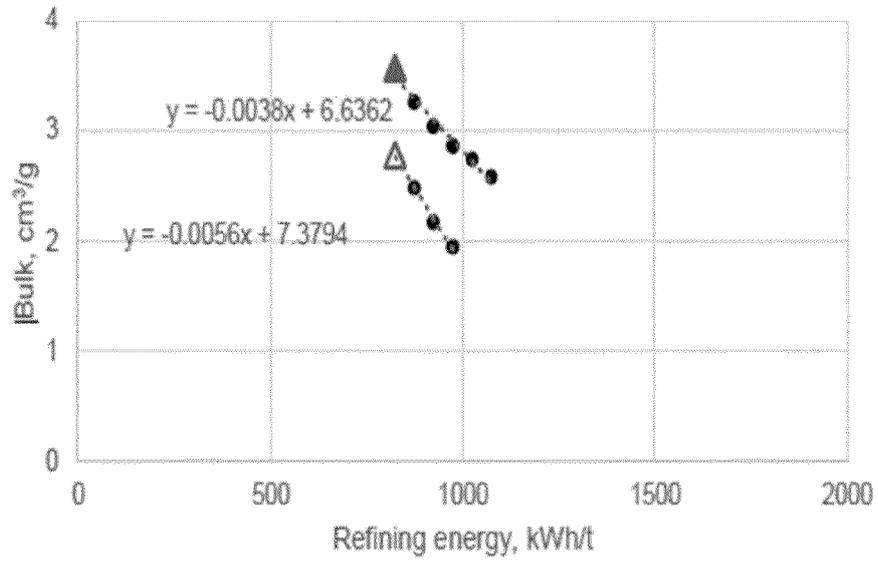


Fig. 4a

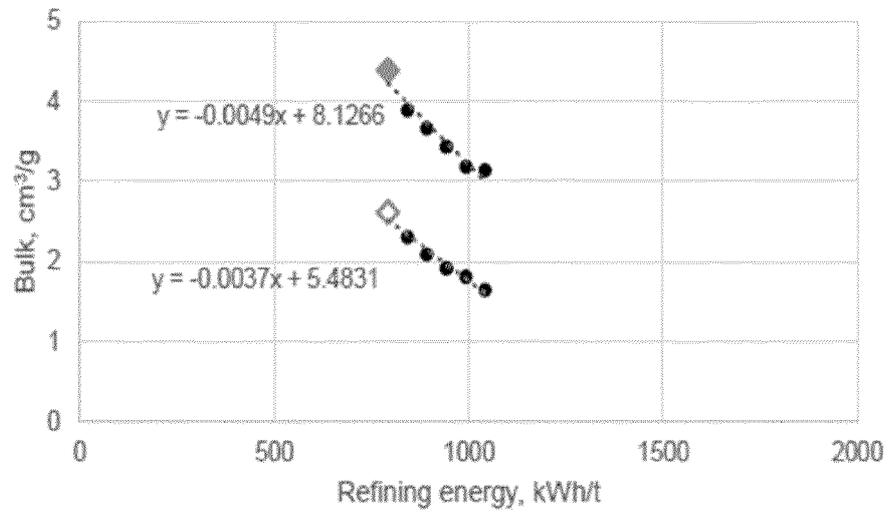


Fig. 4b

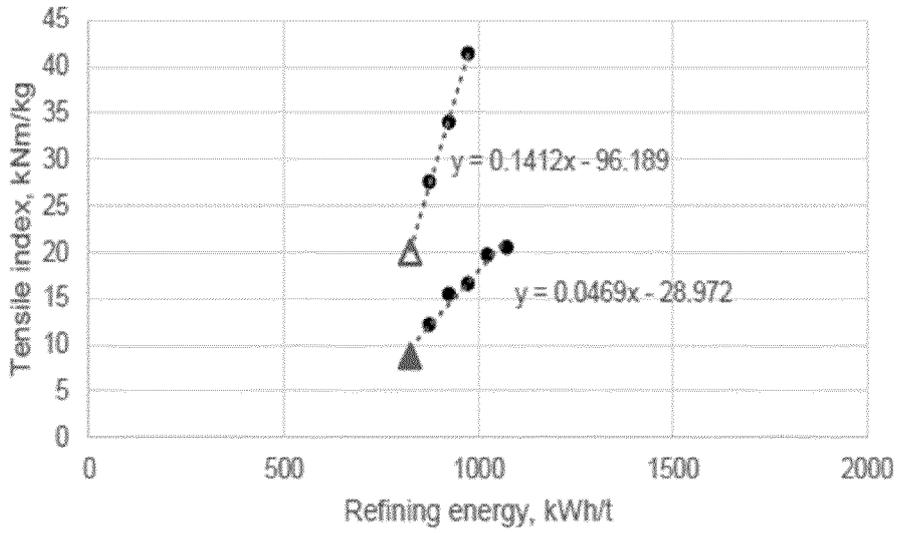


Fig. 5a

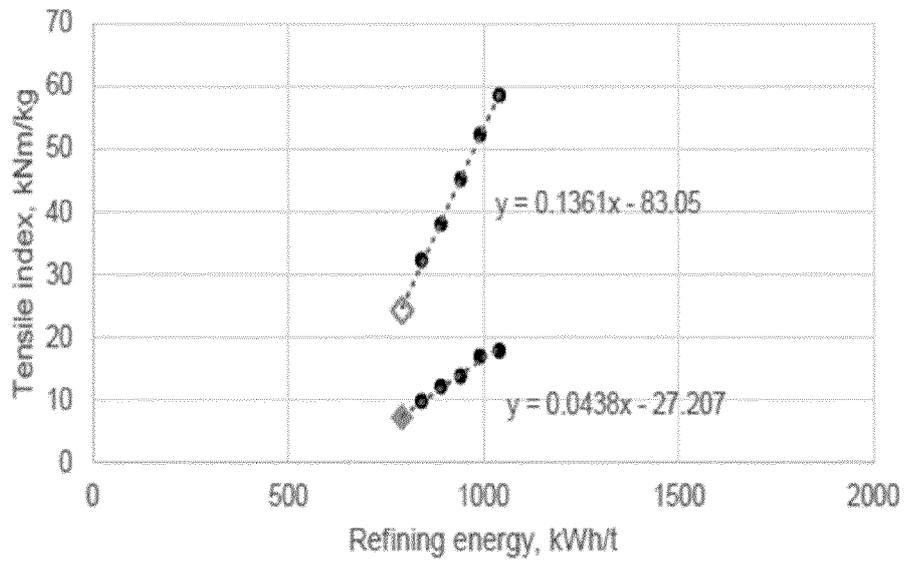


Fig. 5b

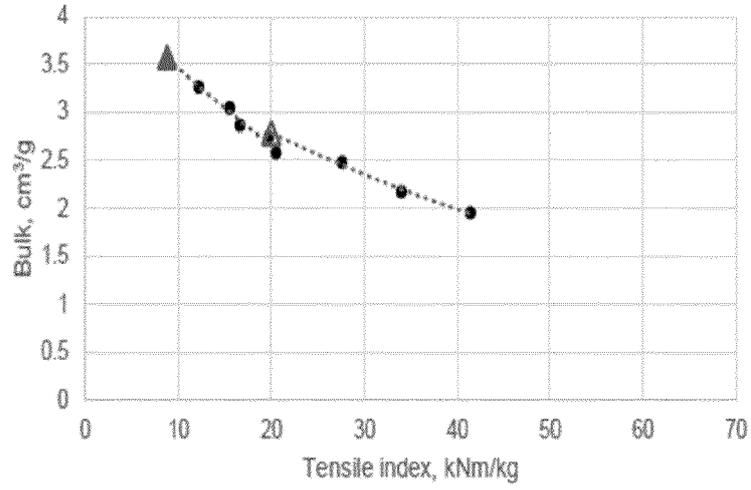


Fig. 6a

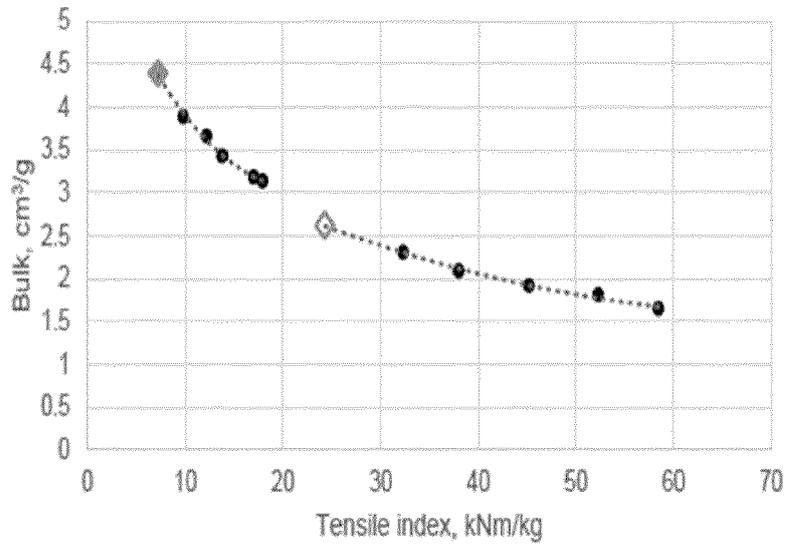


Fig. 6b

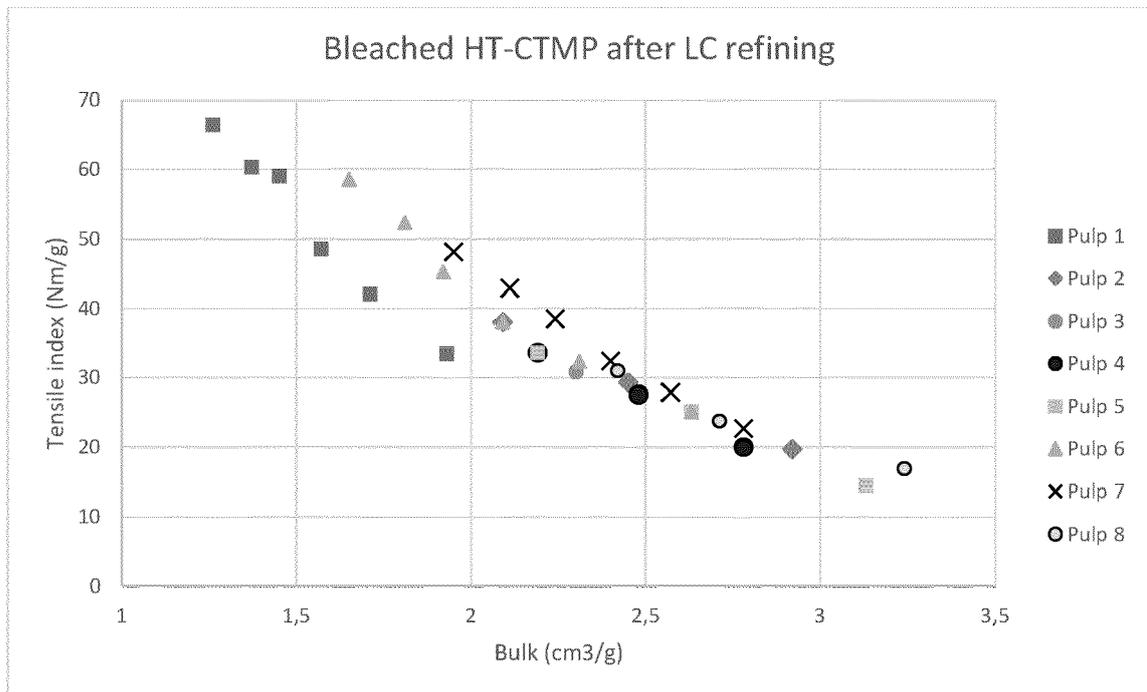


Fig. 7

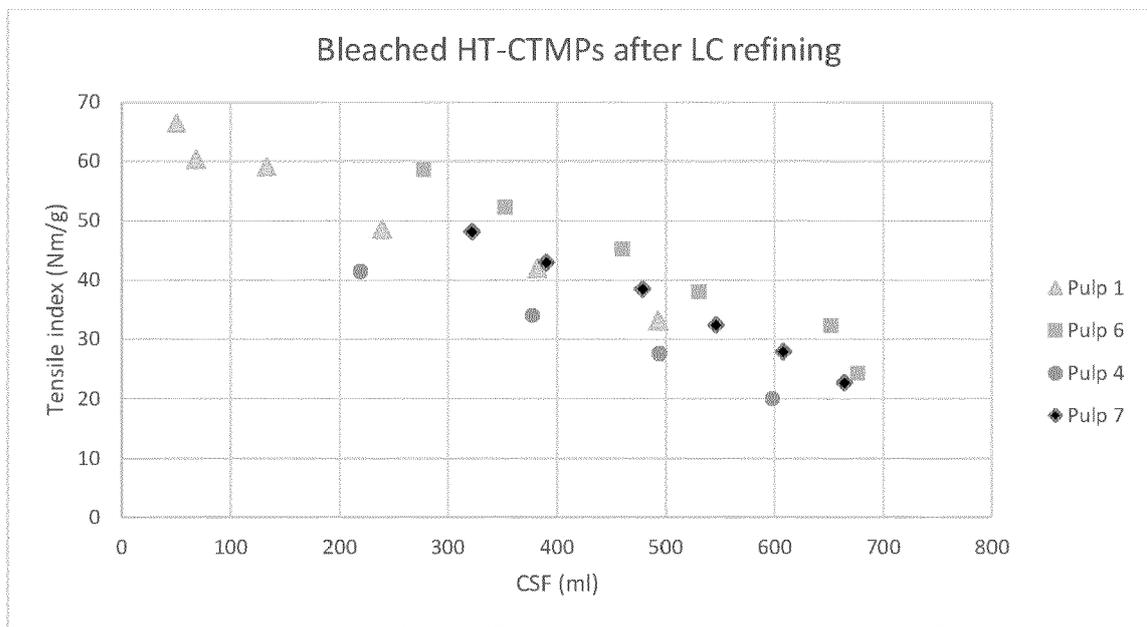


Fig. 8



EUROPEAN SEARCH REPORT

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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	US 4 798 651 A (KOKTA BOHUSLAV [CA]) 17 January 1989 (1989-01-17) * claims 1-23 *	1-14	INV. D21C1/02 D21B1/02 D21H11/02
X	WO 03/000982 A1 (HOLMEN AKTIEBOLAG [SE]; ENGSTRAND PER [SE] ET AL.) 3 January 2003 (2003-01-03) * claims 1-10; example 1 *	1-14	D21B1/14 D21C1/06 D21C9/10 D21B1/021
X	US 4 486 267 A (PRUSAS ZENON C [US]) 4 December 1984 (1984-12-04) * claims 1-16; example 1 * * column 7, line 11 - line 13 *	1-14	
			TECHNICAL FIELDS SEARCHED (IPC)
			D21C D21H D21B
The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 12 May 2023	Examiner Ponsaud, Philippe
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	

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ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.

EP 22 21 5619

5 This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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12-05-2023

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4798651 A	17-01-1989	BR 8801294 A	25-10-1988
		CA 1230208 A	15-12-1987
		EP 0284585 A2	28-09-1988
		ES 2005527 A4	16-03-1989
		FI 881261 A	25-09-1988
		NZ 223929 A	27-03-1990
		PT 87062 A	30-03-1989
		US 4798651 A	17-01-1989
WO 03000982 A1	03-01-2003	AT 312970 T	15-12-2005
		CA 2451155 A1	03-01-2003
		CZ 20033405 A3	16-03-2005
		DE 60208034 T2	24-08-2006
		EP 1407073 A1	14-04-2004
		US 2004231811 A1	25-11-2004
		WO 03000982 A1	03-01-2003
		US 4486267 A	04-12-1984
JP S60110994 A	17-06-1985		
SE 462222 B	21-05-1990		
US 4486267 A	04-12-1984		