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(54) HIGH-BULK CTMP

(57) The present disclosure provides a high temperature chemithermomechanical pulp (HT-CTMP) formed from a mixture of hardwood and spruce wood, wherein

the dry weight ratio of hardwood to spruce wood in said mixture is between 65:35 and 20:80. A method of producing HT-CTMP is also provided.

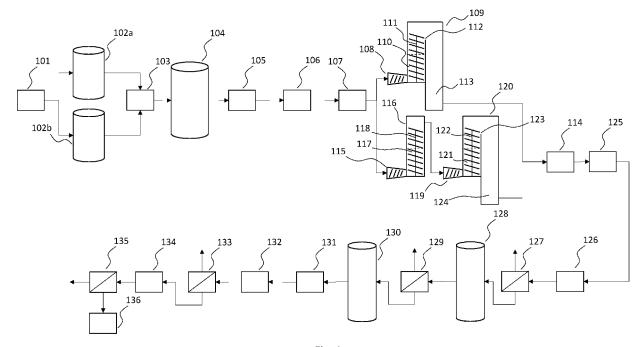


Fig. 1

Description

TECHNICAL FIELD

5 [0001] The present invention relates to the field of chemithermomechanical pulp (CTMP) and the production thereof.

BACKGROUND

[0002] Chemithermomechancial 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 (typically in several steps) 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 steam of relatively high temperature in the heat treatment typically leads to a decrease in the energy input needed during the pressurized defibration step.

SUMMARY

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[0003] It is an objective of the present disclosure to provide a chemithermomechanical pulp (CTMP) of improved properties.

[0004] Accordingly, the present disclosure provides a high temperature chemithermomechanical pulp (HT-CTMP) formed from a mixture of hardwood and spruce wood, wherein the dry weight ratio of hardwood to spruce wood in said mixture is between 65:35 and 20:80.

[0005] The present disclosure further provides a method of forming a high temperature chemithermomechanical pulp (HT-CTMP) comprising the steps of:

- a) mixing hardwood chips and spruce wood chips to obtain a mixture, wherein the dry weight ratio of hardwood to spruce wood in said mixture is between 65:35 and 20:80;
- b) impregnating the chips of the mixture with an impregnation liquid comprising sulfite to obtain impregnated chips;
- c) applying steam having a temperature of at least 150°C to the impregnated chips to obtain pretreated chips; and
- d) defibration of the pretreated chips.

BRIEF DESCRIPTION OF THE DRAWINGS

35 [0006]

Figures 1-3 show exemplary embodiments of a full-scale system for producing HT-CTMP according to embodiments of the present disclosure.

Figure 4 show results obtained in Pilot trial 2 described below.

DETAILED DESCRIPTION

[0007] As a first aspect of the present disclosure, there is provided a high temperature chemithermomechanical pulp (HT-CTMP) formed from a mixture of hardwood and spruce wood, wherein the dry weight ratio of hardwood to spruce wood in said mixture is between 65:35 and 20:80. The hardwood may for example be birch wood.

[0008] 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 at least 150°C.

[0009] In one embodiment, the dry weight ratio of hardwood to spruce wood in said mixture is between 60:40 and 25:75, such as between 60:40 and 45:55.

[0010] In another embodiment, the dry weight ratio of hardwood to spruce wood in said mixture is between 40:60 and 20:80.

[0011] As a second aspect of the present disclosure, there is provided a method of forming a high temperature chemithermomechanical pulp (HT-CTMP) comprising the steps of:

- a) mixing hardwood chips and spruce wood chips to obtain a mixture, wherein the dry weight ratio of hardwood to spruce wood in said mixture is between 65:35 and 20:80;
- b) impregnating the chips of the mixture with an impregnation liquid comprising sulfite to obtain impregnated chips;

- c) applying steam having a temperature of at least 150°C to the impregnated chips to obtain pretreated chips; and d) defibration of the pretreated chips.
- [0012] The chips from step a) are typically washed and then pre-steamed before being impregnated in step b). Embodiments of the washing and pre-steaming as well as other preparatory steps are described in the examples section below
 - **[0013]** In an embodiment of the second aspect, the dry weight ratio of hardwood to spruce wood in said mixture may be between 60:40 and 25:75, such as between 60:40 and 45:55.
 - **[0014]** In another embodiment of the second aspect, the dry weight ratio of hardwood to spruce wood in said mixture may be between 40:60 and 20:80.
 - **[0015]** 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.
 - **[0016]** In step b), the chips may be fed to an impregnation zone comprising the impregnation liquid using a plug screw (or another compressing device) such that the chips expand in the impregnation zone and absorb the impregnation liquid, thereby providing the impregnated chips.

[0017] In another embodiment, step b) comprises:

- feeding the chips to a pre-impregnation zone comprising a pre-impregnation liquid using a plug screw (or another compressing device) such that the chips expand in the pre-impregnation zone and absorb the pre-impregnation liquid, thereby providing pre-impregnated chips; and
- feeding the pre-impregnated chips to an impregnation zone comprising the impregnation liquid using a plug screw (or another compressing device) such that the pre-impregnated chips expand in the impregnation zone and absorb the impregnation liquid, thereby providing the impregnated chips.

[0018] In this embodiment, the temperatures of the pre-impregnation liquid and the impregnation liquid are preferably at least 70°C, such as 70°C-99°C, such as 80°C-99°C. At such temperatures, the viscosity of the liquids is lower, which facilitates the absorption thereof.

[0019] The pre-impregnation liquid is typically water to which NaOH may be added.

- [0020] In one embodiment, the impregnated chips obtained in step b) are transferred to step c) without compressing the impregnated chips. Hence, no plug screw is used for the transfer of the impregnated chips in this embodiment. Instead, the transfer of the impregnated chips may comprise lifting the impregnated chips out of the impregnation liquid using a transport screw and then allowing the impregnated chips to fall into a heating zone in which the steam-based heat-treatment of step c) takes place.
- [0021] The amount of Na₂SO₃ supplied to step b) maybe 10-30 kg, such as 15-25 kg, per dry tonne wood chips supplied to step b).
 - [0022] In one embodiment, less than 10 kg NaOH, such as less than 5 kg NaOH, per tonne dry wood chips is supplied to step b).
 - **[0023]** In one embodiment, the impregnation liquid has a pH below 10.9. Such a pH reflects a relatively low (or no) supply of NaOH.
 - **[0024]** In one embodiment, the temperature of the steam applied in step c) is at least 155°C, such as at least 160°C. An upper limit may be 190°C.
 - [0025] The residence time in step c) is preferably no more than two minutes.
 - **[0026]** The defibration of step d) is typically carried out under pressure.
- [0027] The pulp obtained from step d) may be subjected to refining (such as low consistency refining) and/or bleaching. Embodiments of such refining and/or bleaching are described in the examples section below with reference to figures 1-3.

EXAMPLES

⁵⁰ Pilot trial 1

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[0028] Three batches of wood chips were prepared:

Batch 1 - 100% birch wood chips;

Batch 2 - 30% birch wood chips and 70% spruce chips (percentages on a dry weight basis); and

Batch 3 - 100% spruce wood chips.

[0029] After washing, the chips of each batch were subjected to pre-steaming for 10 minutes using atmospheric steam.

[0030] The chips were then impregnated with an aqueous impregnation liquid (temp. = 40°C) in an impregnation vessel of a pilot plant. The washed and pre-steamed chips were fed to the impregnation vessel using a plug screw such that the chips expanded in the impregnation liquid.

[0031] For batch 1, NaOH, Na₂SO₃ and DTPA were supplied to the impregnation vessel in amounts of 20, 20 and 2 kg per tonne dry chips, respectively.

[0032] For batch 2, Na₂SO₃ and DTPA were supplied to the impregnation vessel in amounts of 20 and 2 kg per tonne dry chips, respectively.

[0033] For batch 3, Na_2SO_3 and DTPA were supplied to the impregnation vessel in amounts of 25 and 2 kg per tonne dry chips, respectively.

[0034] The impregnated chips were then heated by the application of steam having a temperature of 165°C (batches 1 and 2) or 170°C (batch 3). No plug screw was used to transfer the chips from the impregnation to the steaming step. Hence, the chips were transferred to the steaming step without being compressed. The residence time in the steaming step was less than 2 minutes. The pretreated chips from the steaming step were subjected to high consistency defibration/refining such that pulps were obtained. The pulps were divided into sub-batches, which were subjected to different degrees of low consistency (LC) refining. Properties of the pulps were then measured (see table 1 below). Further, sheets were formed from the pulps according to ISO 5269-1 and properties of the sheets were measured.

[0035] Table 1. Pulp and sheet properties. "Deg. of LC ref." means degree of low consistency refining and is the specific energy consumption (kWh/dry tonne wood chips) in the refining step.

Batch (pulp)	Birch/Spruce (wt.%/wt.%)	Deg. of LC ref.	CSF (ml)	SR (°)	Bulk (cm ³ /g)	
1	100/0	0	730*	14.2	3.57	
1	100/0	50	677*	16.4	3.26	
1	100/0	100	600*	20.0	3.04	
1	100/0	150	485*	26.2	2.86	
1	100/0	200	388*	33.1	2.74	
1	100/0	250	315*	38.9	2.58	
2	30/70	0	697	N/A	4.60	
2	30/70	50	684	N/A	4.44	
2	30/70	100	669	N/A	4.09	
3	0/100	0	670*	16.7	N/A	
3	0/100	50	672*	16.6	N/A	
3	0/100	100	613*	19.2	N/A	
3	0/100	150	595*	20.2	3.34	
3	0/100	200	610*	19.5	3.22	
3	0/100	250	612*	19.3	3.25	
* Estimated based on measured SR value						

[0036] As shown in table 1, the 30/70 mixture (batch 2) resulted in considerably higher (about 1 cm³/g higher) bulk values than 100% birch (batch 1) at comparable freeness values. The 100% spruce batch (batch 3) does not indicate that spruce wood would have such an effect in mixture with birch wood. Hence, the results obtained with the mixture of batch 2 could not have been predicted based on experimentation on the individual starting materials alone.

Pilot trial 2

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[0037] Two batches of wood chips were prepared:

Batch 1 - 100% birch wood chips; and

Batch 2 - 55% birch wood chips and 45% spruce chips (percentages on a dry weight basis).

[0038] After washing, the chips of each batch were subjected to pre-steaming.

[0039] The chips were then impregnated with an aqueous impregnation liquid comprising NaOH, Na_2SO_3 and DTPA in an impregnation vessel of a pilot plant. The washed and pre-steamed chips were fed to the impregnation vessel using a plug screw such that the chips expanded in the impregnation liquid. For both batches, NaOH, Na_2SO_3 and DTPA were supplied to the impregnation vessel in amounts of 10, 20 and 2 kg per tonne dry chips, respectively.

[0040] The impregnated chips were then heated by the application of steam having a temperature of 165°C. No plug screw was used to transfer the chips from the impregnation to the steaming step. Hence, the chips were transferred to the steaming step without being compressed. The residence time in the steaming step was less than 2 minutes. The pretreated chips from the steaming step were subjected to high consistency (HC) defibration/refining such that pulps were obtained. Pulp samples from batch 1 were taken after 667 and 1101 kWh/tonne dry chips of HC refining. The CSF values of these batch 1 pulp samples were 719 and 610 ml, respectively. Pulp samples from batch 2 were taken after 684 and 1049 kWh/tonne dry chips of HC refining. The CSF values of these batch 2 pulp samples were 691 and 536 ml, respectively.

[0041] Sheets were formed from the pulp samples according to ISO 5269-1 and properties (bulk, tensile index) of the sheets were measured. The results are presented in Fig. 4, which shows that at a given bulk value (e.g. 4.5 cm³/g), batch 2 (i.e. 45% spruce) gives much higher tensile strength.

Exemplary embodiment of a full-scale system for producing HT-CTMP

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[0042] Figures 1-3 illustrate exemplary embodiments of a full-scale system for producing HT-CTMP.

[0043] A chipper 101 is used to prepare chips from hardwood (e.g. birch wood) and spruce wood. It may be preferred to prepare hardwood chips that are relatively short, such as < 20 mm, to aid impregnation. Spruce wood chips are generally easier to impregnate and can hence be longer, such as 22-24 mm. However, the spruce chips may also have the same length as the hardwood chips. The settings of a conventional wood chipper can be adjusted to achieve desired chip lengths. Shorter chips from such a chipper are also thinner.

[0044] The hardwood chips and the spruce wood chips from the chipper 101 are stored in a hardwood chips silo 102a and spruce wood chips silo 102b, respectively. A chips mixing system 103 is arranged downstream the silos 102a, 102b to prepare a chips mixture having the desired ratio of hardwood chips to spruce wood chips. This ratio is in the range of 65:35 to 20:80 (based on dry weight).

[0045] 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) at aerobic conditions. 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 extracted in downstream process steps.

[0046] 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.

[0047] Yet another option is to place the maturation silo 104 between the chips washing arrangement 106 and the pre-steaming bin 107 described below. It is also possible to omit the maturation step.

[0048] Before being impregnated, the chips are washed in a chips washing arrangement 106. Upstream the chips 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 moisture content. During cold winter months, ice on the chips is melted in the conditioning device 105, which facilitates the downstream washing and processing. Hence, the conditioning device 105 may be particularly advantageous when there is no upstream maturation silo. In case there is an upstream maturation silo, the conditioning device 105 may be omitted.

[0049] In the chips washing arrangement 106, the chips are typically soaked and agitated in water and then dewatered. 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.

[0050] The steamed chips from the pre-steaming bin 107 are subjected to impregnation in one or two steps.

[0051] In case of one-step impregnation, a plug screw 108 feeds the steamed chips into a reactor 109. The steamed chips, which were compressed in the plug screw 108, expands in a bath of aqueous impregnation liquid no 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 impregnation liquid typically comprises sulfite and optionally alkali. The (expanded and impregnated) chips are lifted from the bath of impregnation liquid no by means of a transport screw 111 and are then allowed to fall over an edge 112 and into steaming area 113 of the reactor 109, in which they are heated by steam having a temperature of at least 150°C. The chips treated in the reactor 109 are transferred to a chips defibrator 114 without flashing off any steam on the way.

[0052] In case of two-step impregnation, a plug screw 115 feeds 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 alkali and optionally sulfite. 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 then feeds the pre-impregnated chips into a reactor 120. The pre-impregnated chips, which were compressed in the plug screw 119, expands 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 impregnation liquid comprises sulfite and optionally some alkali. 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 steaming area 124 of the reactor 120, in which they are heated by steam having a temperature or at least 150°C. The chips treated in the reactor 120 are transferred to the chips defibrator 114 without flashing off any steam on the way.

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[0053] In the chip defibrator 114, the dry matter content may be about 45%-50% (in case there is no plug screw between the steaming area 124 and the chips defibrator 114, the dry matter content may however be as low as 30%). The refined chips from the chips defibrator 114 is subjected to flashing in a steam separator 125 and then pulped in a first pulper 126. The pulp from the first pulper 126 is then treated in 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 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 to the extractives to be adsorbed onto the fibers before the first dewatering press 127.

[0054] The pulp from the first dewatering press 127 has undergone chemical treatment, heat treatment by high temperature steam and mechanical treatment (i.e. defibration/refining) and it thus a HT-CTMP. This pulp may be used in the production of paperboard without further chemical treatment or refining. I may also be subjected to low consistency (LC) refining before being used in paperboard production. Yet another option is to further treat the pulp by bleaching and LC refining as described below.

[0055] 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 the downstream high consistency (HC) bleaching and, if needed, make-up quantities of NaOH and peroxide. MC means 10%-12%. The MC-bleached pulp is treated in a second dewatering press 129 also producing a pressate. The pulp from the second dewatering press 129 has a consistency of about 30%-35% and is subjected to high consistency (HC) bleaching in a HC bleach tower 130 using fresh peroxide and alkali (and optionally a peroxide stabilizer, such as a silicate or a non-silicate stabilizer and/or a chelating agent, such as DTPA or EDTA). The HC-bleached pulp from the HC bleach tower 130 are pulped in a second pulper 131 (residence time: <10 min, such as about 3 min) to produce a pulp having a consistency of about 4%-6%. This pulp is then subjected to low consistency (LC) refining in LC refiners 132. A third dewatering press 133 then separates a third pressate from the LC-refined pulp. The fibers from the third dewatering press 133 are pulped in a third pulper 134 (residence time: <10 min, such as about 3 min) to produce a pulp having a consistency of 2%-4%. Screens 135 are then used to separate a reject from the pulp from the third pulper 134. The separated reject is collected in a reject tank 136.

[0056] The design of the remaining parts of the system depends on if only market pulp is produced (i.e. all CTMP is subjected to flash drying and baling) or if there is an adjacent board-making machine to which at least part of the CTMP is supplied without drying.

[0057] In the former case, which is illustrated in figure 2, the pulp from the screens 135 are cleaned in cleaners 137 to provide cleaned pulp and second reject that is collected in a second reject tank 138. The cleaners 137 are preferably cyclones that separate unwanted heavy particles. 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.

[0058] In the latter case, which is illustrated in figure 3, the pulp from the screens 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.

[0059] To produce (dried) market pulp, a fifth dewatering press 148 produces dewatered fibers and a fifth pressate from 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.

[0060] To use the produced CTMP in the production of paperboard, MC pulp from the MC tower 147 is led to a board-making machine.

Claims

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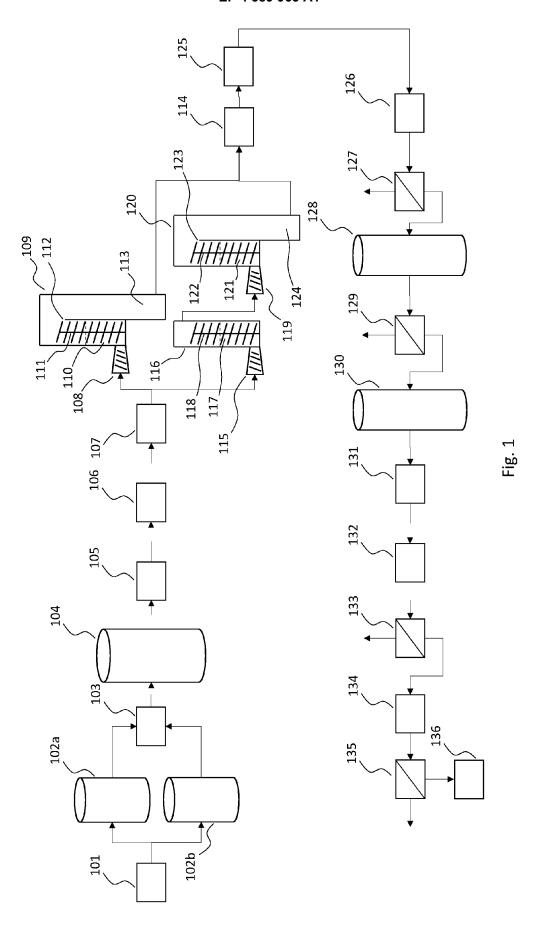
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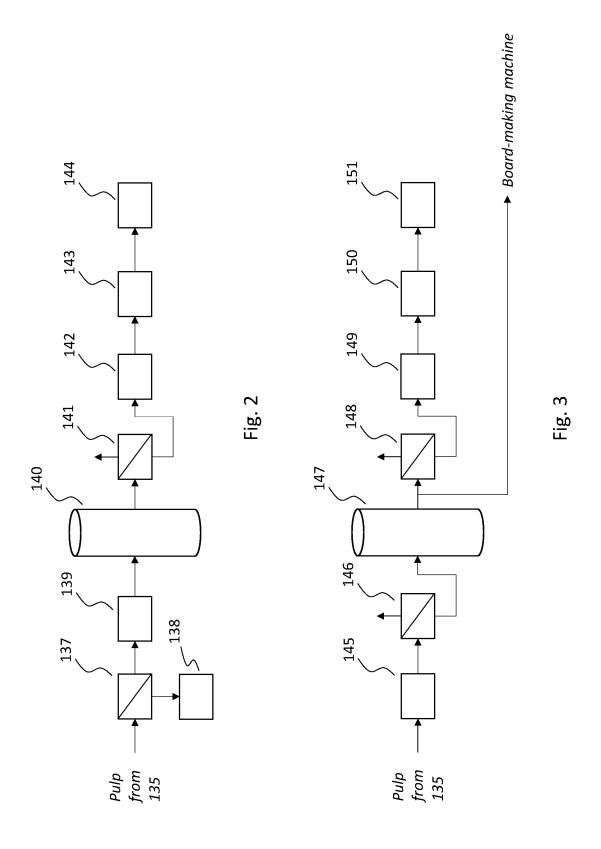
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- 1. A high temperature chemithermomechanical pulp (HT-CTMP) formed from a mixture of hardwood and spruce wood, wherein the dry weight ratio of hardwood to spruce wood in said mixture is between 65:35 and 20:80.
- 2. The HT-CTMP of claim 1, wherein the dry weight ratio of hardwood to spruce wood in said mixture is between 60:40 and 25:75, such as between 60:40 and 45:55.
- 3. The HT-CTMP of claim 1, wherein the dry weight ratio of hardwood to spruce wood in said mixture is between 40:60 and 20:80.
 - 4. The HT-CTMP of any one of the preceding claims, wherein the hardwood is birch wood.
 - 5. A method of forming a high temperature chemithermomechanical pulp (HT-CTMP) comprising the steps of:
 - a) mixing hardwood chips and spruce wood chips to obtain a mixture, wherein the dry weight ratio of hardwood to spruce wood in said mixture is between 65:35 and 20:80;
 - b) impregnating the chips of the mixture with an impregnation liquid comprising sulfite to obtain impregnated chips;
 - c) applying steam having a temperature of at least 150°C to the impregnated chips to obtain pretreated chips; and d) defibration of the pretreated chips.
 - **6.** The method of claim 5, wherein the dry weight ratio of hardwood to spruce wood in said mixture is between 60:40 and 25:75, such as between 60:40 and 45:55.
 - **7.** The method of claim 5, wherein the dry weight ratio of hardwood to spruce wood in said mixture is between 40:60 and 20:80.
- **8.** The method of any one of claims 5-7 wherein the impregnated chips obtained in step b) are transferred to step c) without compressing the impregnated chips.
 - **9.** The method of any one of claims 5-8, wherein the amount of Na₂SO₃ supplied to step b) is 10-30 kg per dry tonne wood chips supplied to step b).
- 10. The method of claim 9, wherein the amount of Na₂SO₃ supplied to step b) is 15-25 kg per dry tonne wood chips supplied to step b).
 - 11. The method of any one of claims 5-10, wherein less than 10 kg NaOH per tonne dry wood chips is supplied to step b).
- 40 **12.** The method of claim 11, wherein less than 5 kg NaOH per tonne dry wood chips is supplied to step b).
 - **13.** The method of any one of claims 5-12, wherein the temperature of the steam applied in step c) is above 155°C.
- **14.** The method of any one of claims 5-13, wherein the temperature of the impregnation liquid is at least 70°C, such as 70°C-99°C, such as 80°C-99°C.

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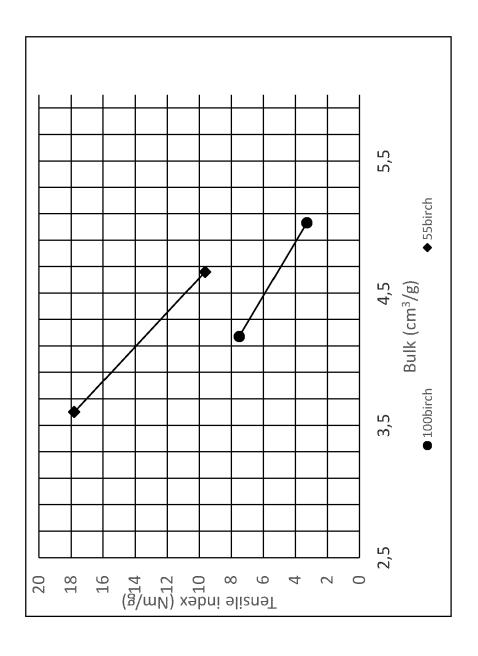


Fig. 4

DOCUMENTS CONSIDERED TO BE RELEVANT

Citation of document with indication, where appropriate,

EP 4 074 892 A1 (TETRA LAVAL HOLDINGS &

ET AL) 21 October 2021 (2021-10-21) * paragraph [0077]; claim 5 *

EP 3 023 539 A1 (UPM KYMMENE CORP [FI])

* paragraphs [0018], [0019], [0058],

FINANCE [CH]) 19 October 2022 (2022-10-19)

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of relevant passages

* claim 3; example 1 *

25 May 2016 (2016-05-25)

* claim 1 *

[0059] *



Category

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EUROPEAN SEARCH REPORT

Application Number

EP 22 21 5611

CLASSIFICATION OF THE APPLICATION (IPC)

INV.

D21C1/02

D21C3/06 D21C9/00

D21H11/02

TECHNICAL FIELDS SEARCHED (IPC)

D21C D21H

Relevant

to claim

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Place of search
Munich
CATEGORY OF CI
X : particularly relevant if Y : particularly relevant if document of the same A : technological backgro O : non-written disclosure

The present search report has been	drawn up for all claims				
Place of search	Date of completion of the search	Examiner			
Munich	11 May 2023	Pon	saud,	Philippe	
ATEGORY OF CITED DOCUMENTS	T: theory or principle underlying the invention				

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FORM 1503 03.82 (P04C01)

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

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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 The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

11-05-2023

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82