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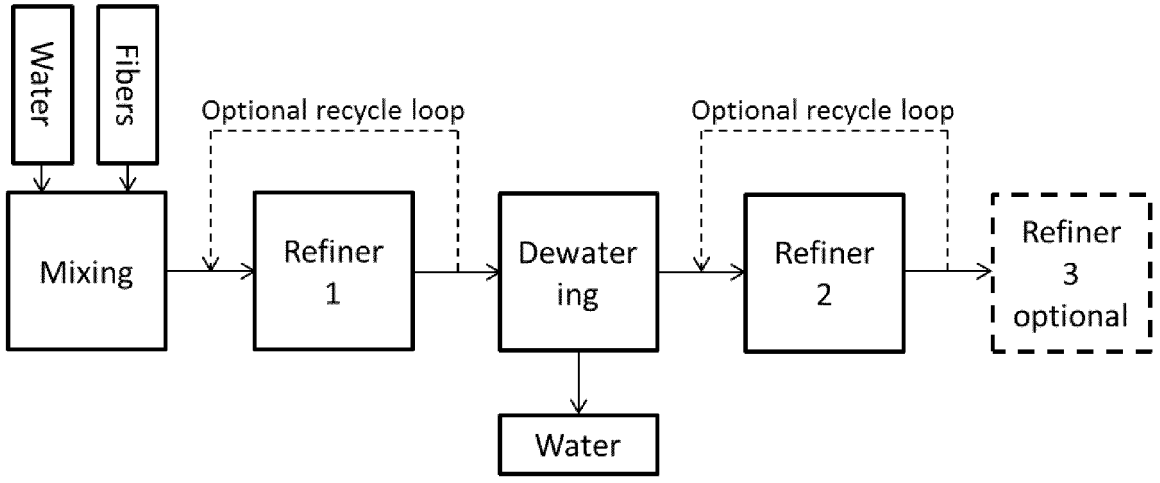
(54) Two-step pulp manufacturing process including a water removal step

(57) Process for the manufacture of aramid pulp, wherein a suspension in water of aramid fibers with an average fiber length of below 15 mm is subjected to a first refining step, the product from the first refining step is subjected to a water removal step, and the product from the water removal step is subjected to a second refining step.

Fig. 1



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Description

[0001] The present invention relates to a process for the manufacture of aramid pulp.

[0002] Processes to prepare pulp from various fiber materials are widely known. Pulp may be prepared by cutting fiber to obtain the desired fiber length followed by fibrillating (also referred to as refining) the fiber to give it a rough or hairy appearance.

[0003] Pulp is defined as a short-cut fibrillated fiber, where filaments are partly loosened from the fiber stem such that a short fiber stem with protruding fibrils and stalks occurs.

Aramid pulp may be derived from aramid fibers which are cut to a length of e.g. 0.5-20 mm, and then subjected to a fibrillation step, wherein the fibers are pulled apart to form the fibrils, whether or not attached to a thicker stem. Pulp of this type may be characterized by a length of, e.g., 0.6-6 mm, and a Schopper-Riegler of 15-85. In some embodiments, the pulp may have a specific surface area (SSA) of 4-20 m^2/g. Within the context of the present specification, the term pulp also encompasses fibrils, i.e., "pulp" which predominantly contains the fibrillated part and little or no fiber stems.

[0004] Pulp can be used in a wide variety of applications e.g. for paper making and as reinforcement material in friction products, sealing products and other plastic or rubber products.

Advantageous are pulps with a high specific surface area (SSA), a high filler retention and a high green strength.

[0005] For the conventional production of aramid pulp, aramid fiber is precut to obtain individual fiber fragments of appropriate short size. The precut fiber is suspended in a liquid and the resulting suspension is fed into a refiner. The refiner fibrillates and further cuts the short fiber fragments.

[0006] Even though processes to prepare aramid pulp are known in the art, there is still room for improvement and to provide alternative processes which result in finely fibrillated pulp with a high specific surface area, a low agglomerate content, a high filler retention and a high green strength.

Such an improved and alternative process is provided by a process for the manufacture of aramid pulp, wherein a suspension in water of aramid fibers with an average fiber length of below 15 mm is subjected to a first refining step, the product from the first refining step is subjected to a water removal step, and the product from the water removal step is subjected to a second refining step.

[0007] A key feature of the process according to the invention is the water removal step between the first and the second refining step which results in the aramid concentration in the second refining step being higher than the aramid concentration in the first refining step. It has been found that this particular combination results in pulp with attractive properties, in particular a pulp with a high degree of fibrillation, resulting in a high specific surface area, a high filler retention and a low number of agglomerates. Paper produced from the pulp manufactured according to the process of the invention has improved mechanical properties.

[0008] In the first refining step the aramid concentration of the suspension of aramid fibers is generally at most 3 wt%, preferably at most 2.5 wt% and more preferably between 1.4 and 2.2 wt%.

In the second refining step the aramid concentration is preferably at least 0.1 wt% higher than that of the suspension provided to the first step, preferably at least 0.2 wt% higher.

Even more preferably, the aramid concentration in the second refining step is at least 0.3 wt% or at least 0.4 wt% or at least 0.5 wt% higher than that of the suspension provided to the first refining step. The aramid concentration of the second refining step can also even be higher, e.g. at least 0.75 wt%, at least 1 wt%, at least 2 wt%, at least 3 wt% or even at least 5 wt% higher than in the first refining step.

[0009] The maximum concentration of the aramid fiber suspension is not critical, in general a maximum of 20 wt% can be mentioned.

[0010] The concentration of aramid refers to the amount of fiber or fibrillated fiber (weight) in the suspension (weight), i.e. grams of dry fiber or pulp per grams of suspension.

[0011] In the context of the present specification aramid refers to an aromatic polyamide which is a condensation polymer of aromatic diamine and aromatic dicarboxylic acid halide. Aramids may exist in the meta- and para-form, both of which may be used in the present invention. The use of aramid wherein at least 85% of the bonds between the aromatic moieties are para-aramid bonds is considered preferred. As typical members of this group are mentioned poly(paraphenylene terephthalamide), poly(4,4'-benzanilide terephthalamide), poly(paraphenylene-4,4'-biphenylenedicarboxylic acid amide) and poly (paraphenylene-2,6-naphthalenedicarboxylic acid amide or copoly(para- phenylene/3,4'-oxydiphenylene terephthalamide). The use of aramid wherein at least 90%, more in particular at least 95%, of the bonds between the aromatic moieties are para-aramid bonds is considered preferred. The use of poly(paraphenylene terephthalamide), also indicated as PPTA is particularly preferred.

[0012] The first suspension is made by weighing the appropriate amount of short cut fiber with an average length ($LL_{0.25}$) of below 15 mm and mixing this amount with the liquid in which refining takes places. Usually, this will be water.

[0013] Preferably, the average length ($LL_{0.25}$) of the short cut fiber is between 1 and 15 mm, preferably between 1.5 and 12, more preferably between 2 and 10 mm and even more preferably between 4 and 8 mm.

[0014] The length of the short cut fiber or pulp refers to the average length weighted length of all fragments above

0.25 mm length ($LL_{0.25}$). The length is determined with the commercially available Pulp Expert(TM) FS (ex Metso) calibrated with commercially available samples of known length.

[0015] The suspension is transported to a refiner. Different transportation devices or installations can be used, e.g. the suspension is provided to the second refining step by way of a pump.

[0016] Refiners are known to the skilled person of the art. Particularly useful for the production of pulp are disc refiners, but also conical or cylindrical refiners can be used. By the term disc refiner is meant a refiner containing one or more pairs of discs that rotate with respect to each other thereby refining ingredients by the shear force between the discs. Suitable types of disc refiners are for instance so-called rotor-stator refiners, where the suspension is brought between 2 or more closely spaced discs which rotate in respect to each other. The surfaces of the discs face each other and are modified to facilitate good mastication and fibrillation.

In the first refining step the short cut fiber is further reduced in length and fibrillated. In the first refiner the average length ($LL_{0.25}$) of the fiber is reduced to a length below 5 mm, preferably below 3.5 mm. In general, a minimum length of 1 mm can be mentioned.

[0017] Subsequently, the suspension leaves the first refiner and is subjected to a water removal step. Water removal means that the water content of the suspension is reduced which yields a suspension with a higher aramid concentration. The water removal can be realized by different means. The means are chosen by the skilled person to match the process mode, i.e. they are chosen to match a batch mode or (semi) continuous process. Examples are (vibrating) belt filters, centrifuges, (screw) presses, (rotating) sieves or the like.

[0018] After the water removal the suspension preferably has an aramid concentration of at least 2 wt%. The aramid concentration can vary between 2 and 7 wt% and can even be higher depending on the degree of water removal.

For further processing the suspension obtained from the second refining step can be diluted to a concentration between 2 and 7 wt%. If so, the suspension is diluted in a tank.

The aramid concentration of the suspension influences the mode of transportation to the second refiner. For example, the suspension can have an aramid concentration in the range of 2 - 3.5 wt%. Such a suspension is relatively liquid and can e.g. be provided to the second refining step by way of a pump.

The suspension provided to the second refining step can also have a higher aramid concentration, e.g. in the range of 3.5 - 7 wt%. A suspension with this higher concentration is more solid and can be provided to the second refining step by way of for example a screw conveyor or belt conveyor.

[0019] The choice of transportation means is not only dependent on the aramid concentration of the suspension. The fiber length and the degree of fibrillation and mastication also plays a role. The more thoroughly the fiber has been refined the higher the concentration of fiber in suspension can be to still give a liquid suspension. It is within the knowledge of the skilled person to select suitable transportation means.

[0020] The aramid-water suspension resulting from the water removal step is further refined in the second refiner. The refiner used in the second refining step can be the same type as in the first refining step or it can be a different type of refiner.

[0021] The pulp produced according to the process of present invention has a higher specific surface area which correlates with the degree of fibrillation. Also the filler retention and the green strength of the pulp produced according to the process of present invention are improved when compared to a process where no water removal has been carried out between the first and second refining step and hence the aramid concentration in both refining steps is more or less equal.

[0022] After the second refiner the average fiber length is approximately 0.5-1.75 mm, more preferably 0.8-1.6 mm, more preferably 0.6-1.5 mm.

The suspension is further processed to provide the desired product.

[0023] In one embodiment the product from the second refining step can be diluted to a suspension with a lower aramid concentration and the diluted product is subjected to a further refining step, i.e. a third refining step. This can be done to obtain pulp of a specific length.

In one embodiment the product from the second refining step is diluted to a suspension with an aramid concentration of at most 2.5 wt.%, in particular at most 2.2 wt.%, more specifically of at most 2 wt.%, and the diluted product is subjected to a further refining step. In general, a minimum concentration of 0.1 wt% or 0.5 wt% can be mentioned.

In one embodiment an additional water removal step is introduced before a thord refining step.

[0024] In one embodiment, the process comprises one or more recycle loops, wherein refiner product is recycled to the same refiner. This means that the suspension resulting from the second refining step is again subjected to refining in the second refiner. Such a procedure is applied in a case where a single pass through two or more refiners is not enough to create the desired properties in terms of fiber length and specific surface area. With a recycle loop, the pulp suspension has the chance to pass several times through the same refiner in order to create an even finer fibrillated pulp.

[0025] It is also possible, that such a recycle loop is applied after the first refining step. Combinations of recycle loops in the first and second refining step are also possible. Furthermore, a process according to this invention can also include recycle loops and a further, third refining step. An additional refining step can again be preceded by an additional dewatering step, similar to the first one.

However, the process always includes at least one water removal step.
The embodiments can be combined as a person skilled in the art sees fit.
Fig. 1 is an overview which shows a possible embodiment of a process according to this invention, including the following process steps: mixing fibers and water to obtain a suspension, a first refining step followed by a dewatering step which is followed by a second refining steps. Optional steps (first and/or second recycle loop in the refiner and an additional refining step) are indicated with broken lines.

[0026] After the last refiner step the suspension comprising water and the fibrillated fiber can be further processed as desired. Suitable processing steps are well-known to the person skilled in the art. Usually, the water of the suspension is removed to form a filter cake of the pulp. Generally, the pulp is then further dried and opened up or fluffed to give the characteristic fluffy pulp.
For example, the product of the last refining step is subjected to a filter step.
In the filter step the water can be removed from the suspension to obtain a filter cake. The product of the filter step can be subjected to a drying step. Optionally, the pulp can be opened.

[0027] The process according to this invention can be carried out in batch mode, in semi-batch mode or continuously. The installation including mixing tanks, transportation means, refiners and water removal equipment is adjusted according to the chosen manner.

[0028] Typically, pulp produced according to the invention has a specific surface area (SSA) of at least 5 m^2/g, preferably at least 7 m^2/g, more preferably at least 9 m^2/g, at least 11 m^2/g or at least 13 m^2/g.
The filler retention is at least 22%, preferably at least 25%, more preferably at least 27%. Depending on the refining conditions the filler retention can also be much higher, for example at least 50%, at least 60% or preferably at least 70%.
The green strength is at least 0.7 mJ/mm^2, preferably at least 1 mJ/mm^2, more preferably at least 1.2 mJ/mm^2.
Preferably, the pulp has at least a filler retention of 25%, a specific surface area of at least 11 m^2/g and a green strength of at least 1 mJ/mm^2.
The pulp produced according to the process of present invention is particularly suited to produce paper, but also to be used in friction applications (often as substitution for asbestos) and gaskets.

[0029] The various embodiments and preference described herein can be combined as will be clear to the person skilled in the art.

[0030] The invention is further explained by the following non-limiting examples.

Examples

[0031] In the present invention the following measuring methods are used:

The specific surface area SSA [m^2/g] is determined by the BET specific surface area method, wherein the adsorption of nitrogen is measured with the aid of a Tristar 3000 apparatus manufactured by Micrometrics. Before the BET-measurement the samples were dried at 200 °C for 30 minutes, under flushing with nitrogen.

[0032] The length weighted length $LL_{0.25}$ [mm] is determined by the Pulp Expert™ FS apparatus (ex Metso) which was calibrated with samples of pulp with known lengths (commercially available). The length weighted length $LL_{0.25}$ [mm] is a length-weighted average length wherein particles are included having a length > 250 μm, i.e. > 0.25 mm.

[0033] The kaolin filler retention [%] is determined as described in the following:

A mixture of 97 wt.-% Kaolin (Laude SP20) and 3 wt.-% of the sample to be tested (i.e. pulp) is prepared on a high-speed vertical mixer. 20 g of said mixture are sieved on a riddle sifter device using a 250 mesh sieve. The remaining material on the sieve given as percentage of the initial amount is determined and is the kaolin filler retention [%].

[0034] The green strength [mJ/mm^2] is determined as described in the following: A mixture of 97 % Kaolin (Laude SP20) and 3 % of the sample to be tested is prepared on a high speed vertical mixer. 10 g of said mixture are molded at 70 bar to a rod with a thickness between 7.5 and 11 mm and a width of 15 mm. The rod is fractured on a pendulum ram impact testing device perpendicular to its main axis and the areal-specific energy, which is necessary for said fracture, is determined as the green strength in units of [mJ/mm^2].

[0035] The bulk volume V_{bulk} given in [ml/g] is a measure for the density of a pulp/filler mixture and is determined as described in the following: A mixture of 97 % Kaolin (Laude SP20) and 3 % of the sample to be tested is prepared on a high-speed vertical mixer. 30 g of said mixture is poured into a graduated cylinder and the bulk volume V_{bulk} is calculated from the filling volume V_{fill} [ml] according to equation (1):

$$V_{bulk}\ [ml/g] = V_{fill}\ [ml] / 30\ [g] \quad (1).$$

[0036] The jolting volume $V_{jolting}$ given in [ml/g] is a measure for the density of a pulp/filler mixture under a certain weight and is determined as described in the following: After determination of the bulk volume, a jolt piston (50 gram) is applied carefully into the graduated cylinder on the surface of the 30 g sample, of which the bulk volume was determined, and the cylinder is fixed on a jolter. The jolting volume $V_{jolting}$ is calculated from the filling volume after 250 strokes, $V_{fill(250)}$ [ml], according to equation (2):

$$V_{jolting}\ [ml/g] = V_{fill(250)}\ [ml] / 30\ [g] \quad (2).$$

[0037] The Schopper-Riegler value (SR, as SR number) is a measure for the dewatering properties of the pulp and is determined as described in the following: 2 g (dry weight) of never dried pulp fibers were dispersed in 1 L water during 250 beats in a Lorentzen and Wettre disintegrator. A well-opened sample is obtained. The Schopper Riegler (SR) value is measured using a Büchel SR apparatus.

[0038] The Domas (DO) value indicates the number of agglomerates in the pulp which are larger than the indicated size (e.g. with an equivalent diameter larger than 150 or 400 μm). Often such agglomerates (clumps, pills and stringers) are non-opened or non-fibrillated particles. The Domas value is determined as described in the following: 10 g of pulp are transferred into 1 L of water and placed in a Lorentzen and Wettre disintegrator and suspended. The suspension is transferred to a Pulmac Masterscreen (screen size of 150 μm) which has a moistened black filter paper (Ählstrom, black, 20.5 cm diameter) placed in the filter unit. Vacuum is applied to filter all of the suspension. The black filter paper is transferred to an oven and dried at 105ºC for circa 1-2 minutes. The black filter paper is subsequently placed on the scanner (Microtek ScanMaker i900 by PTS) with the objects facing the glass plate of the scanner. The scan result is analyzed with the Domas software (by PTS) which determines the number of objects, surface and distribution of the objects. A Domas number for objects with an equivalent diameter larger than 150 and larger than 400 μm is calculated.

[0039] The paper properties were tested on hand sheets with a diameter of 211 mm which were made of 50% pulp and 50% Twaron® 6 mm fiber (Twaron® 1000) on a British sheet mould. To obtain a grammage of 50 g/m^2 0.91 g of pulp and 0.91 g of 6 mm fiber are opened in 2 Liter of water in a Lorentzen and Wettre disintegrator.

[0040] The mass per unit area (also referred to as grammage) and the density were measured on paper conditioned according to ASTM D1776 option 1. For the calculation of the paper density (g/cm^3) additionally the thickness of the paper was determined to relate the mass to the volume of the paper.

Tensile index (Nm/g) and stiffness index (Nm/g) were measured on dried paper (120° C) wherein sample width is 15 mm, sample length (and clamping distance) is 100 mm, and test speed is 10 mm/min at 21 °C/65% RH conditions. The mechanical property measurements were carried out with an Instron instrument and the TI and SI values are calculated according to ASTM D828 and Tappi T494 om-96. The tensile index is an indication of the maximum tension the paper can stand without tearing. The stiffness index describes the rigidity of the paper, i.e. the extent to which is resists deformation when a force is applied.

Experiment 1 - Process according to the invention including water removal and comparison to a process without water removal

[0041] 2 kg of aramid fibers (6 mm) were suspended in 125 liter of water. The suspension with a aramid fiber concentration of 1.6% was refined in a single pass in a Sprout-Bauer 12" lab refiner. The suspension was then dewatered on a vacuum sieve table until the dewatered fibers reach a weight of 10 kg. This solution with an aramid fiber concentration of 20 wt% was fed to the refiner with a screw feeder. Just before the entrance of the refiner, extra water was added to get a consistency of 5wt% inside the refiner. The fibers were then refined in a single pass in a Sprout-Waldron 12" lab refiner. Two different refiner settings were applied. Half of the solution comprising 5 wt% of aramid (total amount 5kg) was refined with a wide plate gap in the Sprout-Waldron 12" lab refiner to a fiber length longer than 1 mm (sample 1 A) and the other half of the same solution was refined with a narrow plate gap in the Sprout-Waldron 12" lab to reach a fiber length shorter than 1 mm (sample 1 B). The plate distance in the refiner influences the length of the obtained pulp. A wide gap between the refiner discs usually results in longer fibers. For both samples pulp properties were determined (Specific Surface Area, filler retention, green strength, see Table 1).

[0042] To compare the refining process according to the invention to a refining process where no water removal takes place 4 kg of aramid fibers (6 mm) were suspended in 200 liter of water (an aramid concentration of 2 wt%). The suspension was refined by circulating over a Sprout-Bauer 12" lab refiner until a fiber length of ± 1.05 mm was reached. Half of the suspension was then removed from the tank (sample 2A) and the other half was further refined until a fiber

length of ± 0.85 mm (sample 2B). For both samples pulp properties were determined (Specific Surface Area, Filler retention, Green Strength).

Table 1: Pulp properties

Sample	Aramid conc. 1st refiner	Aramid conc. 2nd refiner	Final $LL_{0.25}$ (mm)	SSA (m^2/g)	Filler retention (%)	Green strength (J/mm2)
1A	1.6wt%	5wt%	1.12	9.02	72.90	1.69
1B	1.6wt%	5wt%	0.78	12.68	84.10	1.18
2A	2wt%	No second refining step	1.05	6.01	31.90	1.21
2B	2wt%	2wt%	0.85	7.38	46.50	0.92

[0043] The results show that the dewatering of the slurry and refining the dewatered fibers at a higher consistency than the first step (samples 1 A and 1 B) improves the pulp properties significantly.

Experiment 2 - Comparison of process according to the invention and without water removal

[0044] Another comparison experiment was carried out to obtain pulp with a comparable length weighted length $LL_{0.25}$ from a process with water removal according to the invention and a process where the aramid concentration in the 2nd refining step was the same aramid concentration as in the 1st refining step (no water removal).

[0045] For each of samples C and D, 4 kg of aramid fibers (Twaron 1000, 1680f1000 of 6 mm) were suspended in 250 liter of water. The suspension with an aramid fiber concentration of 1.6% was refined in a Sprout-Bauer 12" lab refiner for ca. 3 minutes.

Sample C (reference process) was further refined in a Sprout-Bauer 12" lab refiner until the pulp particles had a $LL_{0.25}$ of ca. 1 mm.

Sample D (process according to the invention) was pumped to a dewatering filter and dewatered. The fibers were again suspended in 133 liter water, resulting in an aramid concentration of 3%. This suspension was further refined in a Sprout-Bauer 12" lab refiner until the pulp particles had a $LL_{0.25}$ of ca. 1 mm (see table 2)

Table 2: overview of comparison experiment 2

Sample	Aramid conc. 1st refiner	Aramid conc. 2nd refiner	Final $LL_{0.25}$ (mm)
C	1.6wt%	1.6wt%	1 ± 0.05
D	1.6wt%	3wt%	1 ± 0.05

[0046] The properties of pulp samples C and D were determined and are shown in table 3.

Table 3: Pulp properties

Characteristic	Sample C	Sample D	Difference C vs. D (%)
Final $LL_{0.25}$ (mm)	1.04	1.02	
SR°	18	25	+ 39
SSA (m^2/g)	5.7	6.5	+ 14
Domas, > 150μm	1032	180	- 83
Domas, > 400μm	102	30	- 71
Filler retention (%)	24	61	+ 160
Green strength (J/mm^2)	1.28	1.17	- 9
Bulk volume (ml/g)	3.47	3.71	+ 7
Jolting volume (ml/g)	2.00	2.13	+ 7

[0047] The results show that the pulp produced according to the method of the invention has much improved properties even when the fiber length is the same. The filler retention, the specific surface area and the Schopper-Riegler are significantly increased. Also, the amount of agglomerates (Domas) of smaller and larger size is greatly reduced. Therefore, the process of present invention results in a much improved pulp.
To test the properties of paper produced from such pulp, paper sheets were prepared from pulp of sample C and D. The properties of the paper are shown in table 4.

Table 4: paper properties of papers prepared with sample C and D

Characteristic	Sample C	Sample D	Difference C vs. D (%)
Mass per unit area (g/m^2)	55.84	56.91	
Density (g/cm^3)	0.16	0.16	
Tensile index (Nm/g)	0.55	1.27	+ 131
Stiffness index (N/g)	0.3	0.68	+ 127

[0048] Paper produced from pulp produced according to the process of this invention (sample D) has a much improved tensile strength and stiffness. The extent of improvement of the paper properties is surprising.
[0049] The results show that the process of the invention results in improved pulp and also in improved paper products comprising the pulp produced according to present invention.

Claims

1. Process for the manufacture of aramid pulp, wherein a suspension in water of aramid fibers with an average fiber length of below 15 mm is subjected to a first refining step, the product from the first refining step is subjected to a water removal step, and the product from the water removal step is subjected to a second refining step.

2. Process according to claim 1, wherein the suspension provided to the first refining step generally has an aramid concentration of at most 3 wt.%, in particular at most 2.5 wt.%, more specifically between 1.4 and 2.2 wt.%.

3. Process according to claim 1 or 2, wherein the suspension provided to the second refining step has an aramid concentration which is at least 0.1 wt.% higher than that of the suspension provided to the first step, in particular at least 0.2 wt.% higher.

4. Process according to any one of the preceding claims, wherein the suspension provided to the second step has an aramid concentration in the range of 2 - 3.5 wt.%.

5. Process according to claim 4, wherein the suspension is provided to the second refining step by way of a pump.

6. Process according to any one of claims 1-3 wherein the suspension provided to the second step has an aramid concentration of 2 -7 wt%.

7. Process according to claim 6, wherein the suspension is provided to the second step by way of a screw conveyor or belt conveyor.

8. Process according to claim 6 or 7, wherein the product from the second refining step is diluted to a suspension with an aramid concentration of at most 2.5 wt.%, in particular at most 2.2 wt.%, more specifically of at most 2 wt.%, and the diluted product is subjected to a further refining step.

9. Process according to any one of the preceding claims, wherein the process is carried out in batch mode, in semi-batch mode, or continuously.

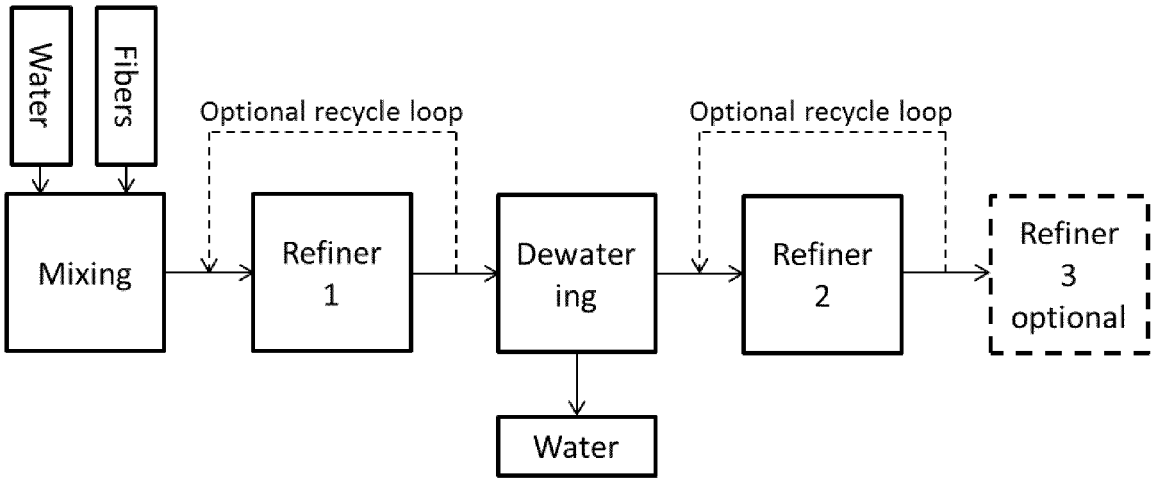
10. Process according to any one of the preceding claims, wherein the process comprises one or more recycle loops wherein refiner product is recycled to the same refiner.

11. Process according to any one of the preceding claims, wherein the product of the last refining step is subjected to

a filter step.

12. Process according to claim 11, wherein the product of the filter step is subjected to a drying step.

Fig. 1


Water
Fibers
Optional recycle loop
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Mixing
Refiner 1
Dewater ing
Refiner 2
Refiner 3 optional
Water

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EUROPEAN SEARCH REPORT

Application Number
EP 13 19 2418

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
A	WO 2007/075575 A2 (DU PONT [US]; LEVIT MIKHAIL R [US]; AMMA ACHIM [US]) 5 July 2007 (2007-07-05) * page 11, line 26 - page 13, line 18; claims 1-15; figures 1/2-2/2 *	1-12	INV. D21B1/12 D21D1/20 D21D1/30 D21H13/26
A	US 2005/284596 A1 (CONLEY JILL A [US] ET AL) 29 December 2005 (2005-12-29) * paragraphs [0065], [0090] - [0095]; claim 1 *	1-12	
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A	GB 938 569 A (DEFIBRATOR AB) 2 October 1963 (1963-10-02) * the whole document *	1	TECHNICAL FIELDS SEARCHED (IPC) D21B D21D D21H

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Munich	23 May 2014	Barker, Stephan

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
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