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(54) **LYOCELL FIBER WITH DECREASED PILL FORMATION**

(57) The present invention provides a lyocell fiber with decreased pill formation while showing a high hemicelluloses content and an increased tendency to fibrillate, as well as a method for producing same and products comprising same.

Figure 1

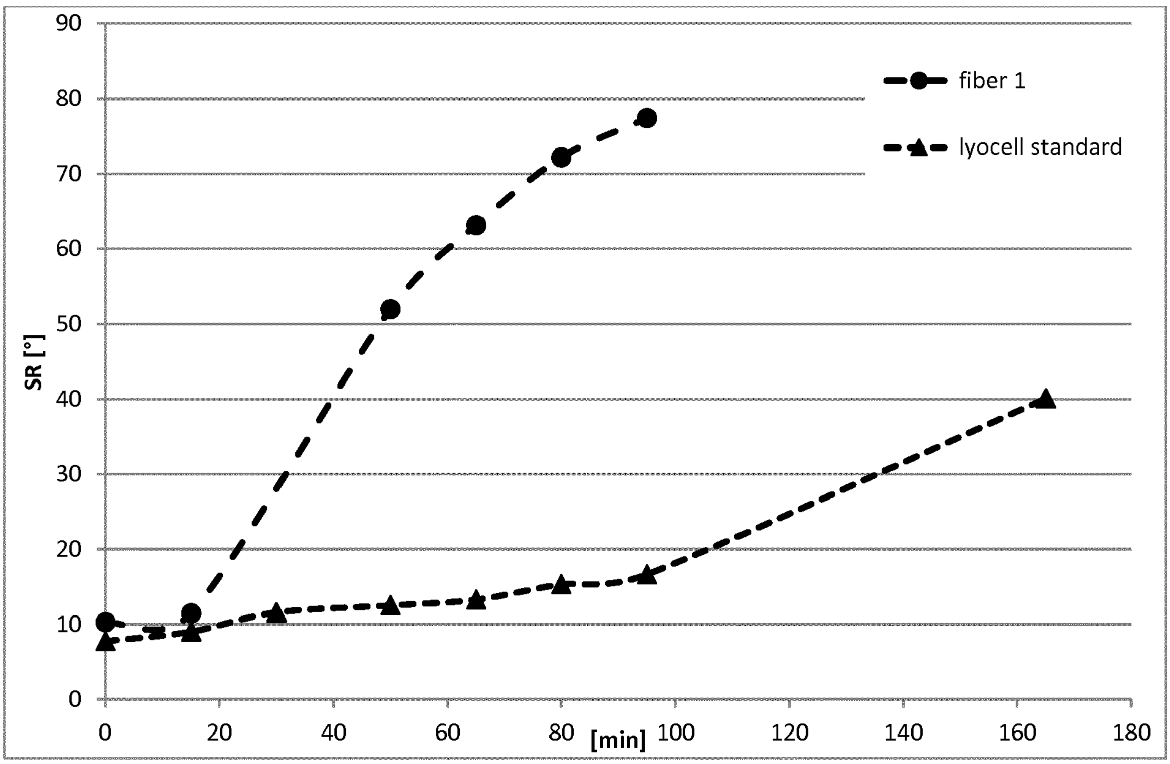

Figure 1: Fibrillation dynamics of two types of fibers

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Description

[0001] The present invention relates to a lyocell fiber with decreased pill formation, a method for producing same as well as products comprising the lyocell fiber.

State of the art:

[0002] Lyocell fibers are known in literature and by experts as fibers with excellent fiber properties (tenacity, elongation and working capacity). The structure of the lyocell fibers leads to outstanding mechanical textile properties reflected in high tenacities in dry and wet state and good dimension stabilities.

[0003] The lyocell fiber is known by experts to show a relatively high fibrillation tendency tracing back to its high fibrillary crystalline structure. This fibrillation can on the one hand be used to generate specific effects on the finished fabric (such as peach skin, silk touch or soft denim). On the other hand this fibrillation can lead to pilling of fabrics and an unsightly optical appearance and touch.

[0004] US 8187422 describes a blend with fibrillated lyocell-microfibers and paper pulp to optimize cleaning performance of the final wipe product resulting in increased opacity and porosity and enhanced softness of the final product. In the patent it is indicated that lyocell staple fibers can be fibrillated in an aqueous medium with low solid content using a disk refiner or a similar device. Usually a chemical pre-treatment is employed for such fibers as to fibrillate the fibers without any chemical pre-treatment would consume enormous amounts of energy and time. US 8187422 discloses that the already fibrillated fibers (obtained from an external supplier) are processed to produce disposable wipes containing 25-75% of these fibrillated fibers.

[0005] The pilling tendency is a well-known challenge in mainly knits made of natural fibers (wholly or blends) with f.e. synthetic fibers. Pilling is formed by fibers sticking out from the yarn on the surface of the fabric, these fibers also tend to fibrillate first. These fibrillated bundles become intertwined and form pills. Its formation is induced by mechanical abrasion in wet conditions. An overview on the state of the art on this topic is given by publications of Schurz et al (Macromolecular Symposia 1994, 83, 1, 273-289), Fink et al (Prog. Polym. Sci. 2001, 26, 1473-1524) and Periyasamy et al (Bangladesh Textile Today, 2012, 4).

[0006] To prevent wet fibrillation of lyocell fibers two different approaches can be followed. Production conditions can be adapted or a chemical cross-linking step can be added to the production process.

[0007] The state of the art for adaptions of production conditions lead only to minor improvements in the fibrillation tendency not being reflected in processing or lead to increased costs or technical effort, which up to now did not lead to a large-scale implementation.

[0008] Adaptations in the production conditions are published in WO 9214871 (pH of spinning bath and washing fields below 8.5 leading to fiber with reduced fibrillation), WO 9735054 (combination of different parameters leads to lower fibrillation - concentration of spinning dope, draw in the air gap, jet hole diameter), WO 9738135 (combination of different parameters leads to lower fibrillation - length of air gap, spinning gas conditions, retention time in air gap), WO 9725462 (addition of an aliphatic alcohol into the spinning bath and washing fields, optionally NaOH can be added as well). The fibrillation tendency of the fiber can also be influenced by additives in the spinning dope. (Chanzy et al (Polymer, 1990, 31, 400-405), Weigel et al (Lenzinger Berichte, 1994, 74, 9, 31-36) or Mortimer et al (J. Appl. Polym. Sci. 1996, 60, 305-316)).

[0009] The second approach - the chemical cross-linking with chemical cross-linking agents or functional reactive dyes - is associated with some disadvantages like additional chemicals and costs, challenges in wastewater treatment, insufficient stability of crosslinking against hydrolysis during the textile process chain. Chemical cross-linking procedures are described in EP 053977, EP 0665904 and EP 0943027.

[0010] WO 2015101543 describes a lyocell fiber with a decreased wet abrasion resistance and a specific location in the Hoeller graph produced from a mixture of two different high alpha content pulps.

[0011] US 8187422 discloses mixtures of lyocell fibers and low DP or standard DP cellulose pulp fibers with a CSF (Canadian Standard Freeness) of 250 ml or less, retaining the CSF values even after drying. The pulp fibers account for 10-75% of total weight of fibers in the mixture. The low DP pulp contains higher amounts of hemicelluloses, but is only added to the mixtures as a low DP pulp, without mentioning any relevance of the hemicellulose content. The fibrillation tendency is measured using the CSF-method (Canadian Standard of Freeness) according to TAPPI Standard T227 om-94.

[0012] US 6706237 discloses that meltblown fibers obtained from hemicelluloses rich pulps show a decreased or reduced tendency to fibrillate. US 8420004 discloses another example of meltblown fibers for producing non-woven fabrics. A similar disclosure is also given in US 6440547, which again refers to meltblown fibers. In these patents the fibers produced show decreased fibrillation tendencies - on the one hand meltblown fibers cannot be compared to fibers produced with the air gap lyocell technology. In these patents fibers produced using this technology are also mentioned - these fibers are produced using lab equipment not allowing the production of lyocell fibers in commercial quality (as for example drawing ratios, after-treatment, production velocities do not reflect scale-up qualities. These fibers not being produced with sufficient after-treatment and with insufficient drawing therefore can be expected to show different structures and properties compared to the fibers produced at production scale.

[0013] Additionally to the steps mentioned before trying to prevent fibrillation, fibrillation which already occurred can be removed by enzymatic and/or mechanical treatment. Specific cellulase enzymes in a controlled environment and/or mechanical treatments (f.e. tumbling) - polish the surface to remove fibrillation. F.e. Carrillo et al (Textile Research Journal 2003, 73, 11, 1024-1030).

[0014] Zhang et al (Polymer Engineering and Science, 2007, 47, 702-706) describe fibers with higher hemicellulose contents. The authors postulate that the fibers tend to show an enhanced fiber fibrillation resistance, lower crystallinity and better dyeability. They also postulate that the tensile strength only decreases insignificantly and that the fiber properties could be even increased further by higher hemicelluloses concentrations in the spinning dope. Zhang et al (Journal of Applied Polymer Science, 2008, 107, 636-641), Zhang et al (Polymer Materials Science and Engineering, 2008, 24, 11, 99-102) disclose the same figures as the paper by Zhang (Polymer Engineering and Science, 2007, 47, 702-706), while Zhang et al (China Synthetic Fiber Industry, 2008, 31, 2, 24-27) describe better mechanical properties. The same authors postulate this same theory in Journal of Applied Science, 2009, 113, 150-156.

[0015] The fibers described in the paper by Zhang et al (Polymer Engineering and Science 2007, 47, 702-706) are produced with lab equipment not allowing the production of lyocell fibers in commercial quality (as for example drawing ratios, production velocities, after-treatment do not reflect scale-up qualities. These fibers not being produced with sufficient after-treatment and with insufficient drawing therefore can be expected to show different structures and properties compared to the fibers produced at production scale.

Object of the present invention

[0016] In view of the increasing demands for fibers based on cellulose raw materials suitable for a broad variety of products, a fiber with an improved, reduced pill formation property is desired. At the same time it would be advantageous if chemical treatments disclosed in the prior art as being advantageous to achieve the desired reduced pill formation could be avoided, as these add costs to the final product and may have detrimental effects on the fibers, such as a poor hydrolysis stability during textile processing, so that the fibers obtained are not always suitable for all desired applications. Therefore the task for the present inventors was to provide fibers that show a reduced or decreased pill formation.

Brief description of the invention

[0017] The present inventors accordingly provide the fiber as defined in claim 1, the method for producing same as described in claim 12 and the use of a specific pulp for preparing the fibers as described in claim 9, as well as products containing same as defined in claim 16. Preferred embodiments are described in the respective subclaims as well as in the specification.

[0018] In particular the present invention provides the following embodiments, which are to be understood as being embodiments for which further explanations are provided below.

1.) Lyocell fiber with decreased pill formation, having a hemicelluloses content of 5 wt.-% or more and a CSF value (8 min) of 350 ml or less.
2.) Lyocell fiber according to embodiment 1, wherein the pill formation measured as the pill count, is at least reduced by 50% after 10 washing cycles compared to a standard Lyocell fiber with the same titer.
3.) Lyocell fiber according to embodiment 1 or 2, wherein the CSF value is 300 ml or less.
4.) Lyocell fiber according to any one of embodiments 1 to 3, wherein the hemicelluloses content is 10 wt.-% or more.
5.) Lyocell fiber according to any one of embodiment s 1 to 4, having a titer of 6.7 dtex or less, such as 2.2 dtex or less, preferably 1.3 dtex or less.
6.) Lyocell fiber according to any one of embodiments 1 to 5, produced from a pulp having a hemicelluloses content of 7 wt.-% or more and 25 wt.-% or less and a xylan content of 6 wt.-% or more.
7.) Lyocell fiber according to any one of embodiment s 1 to 6, wherein the hemicellulose comprises a ratio of C5/xylan to C6/mannan of from 125:1 to 1:3, preferably of from 25:1 to 1:2.
8.) Lyocell fiber according to any one of embodiments 6 or 7, wherein the pulp comprises 5 wt.-% or more xylan, preferably 8 wt.-% or more, more preferably 10 wt.-% or more and/or 3 wt.-% or more mannan, preferably 5 wt.-% or more mannan, and/or 1 wt.-% or less mannan.
9.) Use of a pulp for producing a fiber according to any one of embodiments 1 to 8, wherein the pulp has a hemicelluloses content of 7 wt.-% or more and 25 wt.-% or less.
10.) Use according to embodiment 9, wherein the hemicellulose comprises a ratio of C5/xylan to C6/mannan of from 125:1 to 1:3, preferably of from 25:1 to 1:2.

11.) Use according to any one of embodiments 9 or 10, wherein the pulp comprises 5 wt.-% or more xylan, preferably 8 wt.-% or more, more preferably 10 wt.-% or more and/or 3 wt.-% or more mannan, preferably 5 wt.-% or more mannan, and/or 1 wt.-% or less mannan.

12.) Method for producing the lyocell fiber according to any one of embodiments 1 to 8 using a direct dissolution process.

13.) Method for producing the lyocell fiber according to embodiment 12 using a amine oxide process, where an aqueous solution of the amine oxide and the pulp form a cellulose suspension and a shapeable solution which gets shaped and coagulated in a spin bath obtaining the lyocell fiber after washing and pre-treatment steps.

14.) Method for producing the lyocell fiber according to embodiment 13 using an aqueous tertiary amine oxide, preferably aqueous NMMO.

15.) Method according to any one of embodiments 12 to 14, wherein the pulp comprises 10 wt.-% or more of hemicelluloses.

16.) Product, comprising the lyocell fiber according to any one of embodiments 1 to 8, or the fiber produced according to any one of claims 12 to 15.

17.) Product according to embodiment 16, wherein the product is a non-woven fabric.

18.) Product according to embodiment 16 and/or 17, selected among tissues and wipes.

Brief description of the Figures

[0019] Figure 1 shows the Fibrillation dynamics of two types of fibers. Figures 2 and 3 show the Pilling area and Pilling count after 10 and 15 wash cycles for two types of fibers.

Detailed description of the invention

[0020] The lyocell fiber of the present invention having a decreased pill formation property, can be obtained without need for chemical treatment, such as cross linking or other treatments considered necessary in the prior art. Surprisingly and contrary to the prior art suggestions discussed above, the novel lyocell fibers may be prepared using a pulp having a high hemicellulose content.

[0021] As defined in claim 1 the fiber in accordance with the present invention is a lyocell fiber with an decreased tendency for pill formation without requiring a chemical crosslinking treatment.

[0022] As further defined in the claims, a preferred fiber in accordance with the present invention shows a reduced pill formation, measured as the pill count in accordance with the method as defined in example 3, of at least 50%, preferably at least 60%, more preferably at least 70%, such as up to 80%, compared to a standard Lyocell fiber with the same titer. The standard fiber as referred to herein is a Lyocell fiber produced from a pulp as defined in example 1 as Standard Lyocell pulp.

[0023] The lyocell process is well known in the art and relates to a direct dissolution process of cellulose wood pulp or other cellulose-based feedstock in a polar solvent (for example N-methylmorpholine N-oxide [NMMO, NMO] or ionic liquids). Commercially, the technology is used to produce a family of cellulose staple fibers (commercially available from Lenzing AG, Lenzing, Austria under the trademark TENCEL® or TENCEL™) which are widely used in the textile and nonwoven industry. Other cellulose bodies from lyocell technology have also been produced.

[0024] According to this method the solution of cellulose is extruded in a so called dry-wet-spinning process by means of a forming tool and the moulded solution is guided for example over an air gap into a precipitation bath, where the moulded body is obtained by precipitation of the cellulose. The molding is washed and optionally dried after further treatment steps.

[0025] Such lyocell fibers are well known in the art and the general methodology to produce same is for example disclosed in US 4,246,221 and its analytics in the BISFA (The International Bureau for the Standardization of Man-Made Fibers) publication *"Terminology of Man-Made Fibres",* 2009 edition. Both references are included herewith in their entirety by reference.

[0026] The term lyocell fiber as employed herein defines a fiber obtained by this process, as it has been found that fibers in accordance with the present invention differ greatly from fibers for example obtained from a meltblown process, even if using a direct dissolution process of cellulose wood pulp or other cellulose-based feedstock in a polar solvent (for example N-methylmorpholine N-oxide [NMMO, NMO] or ionic liquids) in order to produce the starting material.

[0027] The term hemicelluloses as employed herein refers to materials known to the skilled person which are present in wood and other cellulosic raw material such as annual plants, i.e. the raw material from which cellulose typically is obtained. Hemicelluloses are present in wood and other plants in form of branched short chain polysaccharides built up by pentoses and/or hexoses (C5 and / or C6-sugar units). The main building blocks are mannose, xylose, glucose, rhamnose and galactose. The back bone of the polysaccharides can consist of only one unit (f.e. xylan) or of two or more units (e.g. mannan). Side chains consist of arabinose groups, acetyl groups, galactose groups and O-acetyl groups as well as 4-O-methylglucuronic acid groups. The exact hemicellulose structure varies significantly within wood species. Due to the presence of sidechains hemicelluloses show much lower crystallinity compared to cellulose. It is well known that mannan predominantly associates with cellulose and xylan with lignin. In sum, hemicelluloses influence the hydrophilicity, the accessibility and degradation behavior of the cellulose-lignin aggregate. During processing of wood and pulp, side chains are cleaved off and the degree of polymerization is decreased. The term hemicelluloses as known by the skilled person and as employed herein comprises hemicelluloses in its native state, hemicelluloses degraded by ordinary processing and hemicelluloses chemically modified by special process steps (e. g. derivatization) as well as short chain celluloses and other short chain polysaccharides with a degree of polymerization (DP) of up to 500.

[0028] Standard lyocell fibers are currently commercially produced from high quality wood pulps with high α-cellulose content and low non-cellulose contents such as hemicelluloses. Commercially available lyocell fibers such as TENCEL™fibers produced from Lenzing AG, show excellent fiber properties for non-wovens and textile applications. As mentioned in the patents referred to above, if a high fibrillation resistance (and accordingly a decreased pill formation) is required these lyocell fibers are either chemically treated using cross-linking agents, and/or adaptions of production conditions are done, being cost and/or technically intensive, or after-treatments (mechanical, enzymes) are employed to remove already generated fibrils.

[0029] A decrease pill formation is known to the expert as decreased pill count (number of pills per dm^2) or decreased pill area (mm^2 per dm^2).

[0030] The lyocell fiber presented in this invention show, in contrast to the presumption in the prior art a reduced pill formation despite showing an increased fibrillation tendency. The inventors assume that the fibrils of the novel fibers described herein are stiffer compared to standard lyocell fibers and therefore do not form bundles and get intertwined with fibers sticking out on the surface of the yarns and form pills. These fibrils break off more easily due to their stiff nature during mechanical stress in the processing so that pill formation is significantly reduced.

[0031] The present invention overcomes the shortcomings of the state of the art by providing lyocell fibers as described herein.

[0032] Preferably these are produced from hemicellulose-rich pulps with a hemicelluloses content of at least 7 wt.-%. Contrary to the disclosure in the prior art discussed above, such high hemicellulose content surprisingly, for lyocell fibers, gives rise to a combination of an increased tendency to fibrillate while showing a decreased pill formation property. Accordingly the present invention surprisingly achieves the tasks as outlined above while using a cellulose based raw material with a higher hemicelluloses content, as compared for standard lyocell fibers. Since the present invention does not require the use of chemical treatments to achieve the desired decreased pill formation the process concerns discussed above can be overcome.

[0033] The pulps preferably employed in the present invention do show as outlined herein a high content of hemicelluloses. Compared with the standard low hemicellulose content pulp employed for the preparation of standard lyocell fibers the preferred pulps employed in accordance with the present invention do show also other differences, which are outlined below.

[0034] Compared with standard pulps the pulps as employed herein display a more fluffy appearance, which results after milling (during preparation of starting materials for the formation of spinning solutions for the lyocell process), in the presence of a high proportion of larger particles. As a result the bulk density is much lower, compared with standard pulps having a low hemicellulose content. This low bulk density requires adaptions in the dosage parameters (f.e. dosage from at least 2 storage devices). In addition the pulps employed in accordance with the present invention are more difficult to impregnate with NMMO. This can be seen by evaluating the impregnating behavior according to the Cobb evaluation. While standard pulps do show a Cobb value of typically more than 2.8 g/g (determined according to DIN EN ISO 535 with the adaptation of employing an aqueous solution of 78% NMMO at 75° C with an impregnation time of 2 minutes), the pulps employed in the present invention do show Cobb values of about 2.3 g/g. This requires an adaptation during spinning solution preparation, such as increased dissolution time (f.e. explained in WO 9428214 and WO 9633934) and/or temperature and/or increased searing during dissolution (f.e. WO9633221, WO9805702 and WO 9428217). This ensures the preparation of a spinning solution enabling the use of the pulps described herein in standard lyocell spinning processes.

[0035] In one preferred embodiment of the present invention the pulp employed for the preparation of the lyocell products, preferably fibers, as described herein, has a scan viscosity in the range of from 300-440 ml/g, especially 320-420 ml/g, more preferably 320 to 400 ml/g. The scan viscosity is determined in accordance with SCAN-CM 15:99 in a cupriethylenediamine solution, a methodology which is known to the skilled person and which can be carried out on commercially available devices, such as the device Auto PulpIVA PSLRheotek available from psl-rheotek. The scan viscosity is an important parameter influencing in particular processing of the pulp to prepare spinning solutions. Even if two pulps seem to be of great similarity as raw material for the lyocell-process, different scan viscosities will lead to completely different behaviour different during processing. In a direct solvent spun process like the lyocell-process the pulp is dissolved in NMMO as such. No ripening step exists comparable to the viscose process where the degree of polymerization of the cellulose is adjusted to the needs of the process. Therefore, the specifications for the viscosity of the raw material pulp typically are within a small range. Otherwise, problems during production may arise. In accordance with the present invention it has been found to be advantageous if the pulp viscosity is as defined above. Lower viscosities compromise mechanical properties of the lyocell products. Higher viscosities in particular may lead to the viscosity of the spinning dope being higher and therefore, spinning will be slower. With a slower spinning velocity lower draw ratios will be attained, which significantly alters the fiber structure and its properties (Carbohydrate Polymers 2018, 181, 893-901; Structural analysis of loncell-F fibres from birch wood, Shirin Asaadia; Michael Hummel; Patrik Ahvenainen; Marta Gubitosic; Ulf Olsson, Herbert Sixta). This will require process adaptations and will lead to a decrease in mill capacity. Employing pulps with the viscosities as define here enables smooth processing and production of high quality products.

[0036] As employed herein the terms lyocell process and lyocell technology relate to a direct dissolution process of cellulose wood pulp or other cellulose-based feedstock in a polar solvent (for example N-methylmorpholine N-oxide [NMMO, NMO] or ionic liquids). Commercially, the technology is used to produce a family of cellulose staple fibers (commercially available from Lenzing AG, Lenzing, Austria under the trademark TENCEL® or TENCEL™) which are widely used in the textile and nonwoven industry. Other cellulose bodies from lyocell technology have also been produced. According to this method the solution of cellulose is usually extruded in a so called dry-wet-spinning process by means of a forming tool and the moulded solution gets for example over an air gap into a precipitation bath, where the moulded body is obtained by precipitation of the cellulose. The moulding is washed and optionally dried after further treatment steps. A process for production of lyocell fibers is described, for instance, in US 4,246,221, WO 93/19230, WO95/02082 or WO97/38153. As far as the present application discusses the drawbacks associated with the prior art and the unique properties for novel products as disclosed and claimed herein in the context of using laboratory equipment (in particular in the prior art) or (semi-commercial) pilot plants and commercial fiber spinning units, the present invention is to be understood to referring to larger scale plants/units, which may be considered as follows concerning their respective production capacity:

semi-commercial pilot plant: about1 kt/a

commercial unit >30 kt/a

[0037] As already outlined above, Zahng et al (Polym. Engin. Sci., 2007, 47, 702-706) describe fibers with high hemicellulose contents. The authors postulate that the fibers received tend to show an enhanced fiber fibrillation resistance.

[0038] The fibrillation tendencies of the fibers are analyzed according to ISO 5267-1:1999 - determination of drainage - Part 1: Schopper-Riegler (SR) method and T227 om-94 Canadian standard method (CSF). The SR method provides a degree of drainage velocity of a diluted cellulose fiber suspension. The CSF relates to the freeness of the pulp and in the understanding of this invention is measured after 8 min mixing time. Surprisingly the present invention provides fibers with completely different properties as with the higher hemicelluloses content the fibrillation tendency increases, while at the same time pill formation is reduced. One possible explanation for these contrasting findings may be the fact that the fibers in accordance with the present invention are fibers produced using large scale production equipment, while the fibers described in the paper by Zhang et al are produced with lab equipment not allowing the production of lyocell fibers in commercial quality (as for example drawing ratios, production velocities, after-treatment do not reflect scale-up qualities). The fibers, not being produced with sufficient after-treatment and with insufficient drawing therefore show different structure and properties compared to the fibers produced at production scale at titers reflecting market applications.

[0039] The content of hemicelluloses may be adjusted according to procedures known in the art. The hemicellulose may be the hemicelluloses originating from the wood from which the pulp is obtained, it is however also possible to add individual hemicelluloses depending on the desired fiber properties from other sources to high purity cellulose with a low original hemicellulose content. The addition of individual hemicelluloses may also be employed to adjust the composition of the hemicelluloses content, for example to adjust the ratio of hexoses to pentoses.

[0040] The pulp enabling the preparation of the fibers in accordance with the present invention preferably shows a ratio of C5/xylan to C6/mannan of from 125:1 to 1:3, preferably in the range of 25:1 to 1:2.

[0041] The hemicellulose content, independent or in combination with the above disclosed ratio, may be 7 wt.-% or more, preferable 10 wt.-% or more and in embodiments up to 25 wt.-% or even 30 wt.-%. In embodiments the xylan content is 5 wt.-% or more, such as 8 wt.-% or more, and in embodiments 10 wt.-% or more. In embodiments, either in isolation or in combination with the above mentioned hemicelluloses and/or xylan contents, the mannan content is 3 wt.-% or more, such as 5 wt.-% or more. In other embodiments the mannan content, preferably in combination with a high xylan content as defined above, may be 1 wt.-% or less, such as 0.2 wt.-% or 0.1 wt.-% or less.

[0042] As mentioned above, the hemicelluloses content in the fibers of the present invention generally is higher, as compared to standard lyocell fibers. Suitable contents are 7 wt.-% or more and up to 30 wt.-%. Preferably the fiber in accordance with the present invention shows a ratio of C5/xylan to C6/mannan of from 125:1 to 1:3, preferably in the range of 25:1 to 1:2. As regards the xylan and/or mannan content the above provided embodiments described in relation with the pulp are applicable also for the fiber as such.

[0043] The fibers in accordance with the present invention typically have a titer of 9 dtex or less, such as 3.3 dtex or less, such as 2.2 dtex, depending on the desired application. If the fiber is intended to be used in non-woven applications a titer of 1.7 dtex or less, such as 1.3 dtex or less typically is suitable. However, the present invention also covers fibers with much lower titers, with suitable lower limits for titers being 0.5 dtex or higher, such as 0.8 dtex or higher, and in embodiments 1.3 dtex or higher. These upper and lower values as disclosed here define ranges of from0.5 to 9 dtex, and including all further ranges formed by combining any one of the upper values with any one of the lower values.

[0044] The fiber in accordance with the present invention may be prepared using lyocell technology employing a solution of cellulose and a spinning process employing a precipitation bath according to standard lyocell processes, known to the skilled person.

[0045] The fibers in accordance with the present invention may be employed for a variety of applications, including yarn formation and the preparation of knitted textiles, but also for the production of non-woven fabrics.

[0046] As far as the present application refers to parameters, such as crystallinity, scan viscosity etc., it is to be understood that same are determined as outlined herein, in the general part of the description and/or as outlined in the following examples. In this regard it is to be understood that the parameter values and ranges as defined herein in relation to fibers refer to properties determined with fibers derived from pulp and containing only additives, such as processing aids typically added to the dope as well as other additives, such as matting agents (TiO_2, which often is added in amounts of 0.75 wt.-%), in a total amount of up to 1 wt.-% (based on fiber weight). The unique and particular properties as reported herein are properties of the fibers as such, and not properties obtained by addition of particular additives and/or post spinning treatments (such as fibrillation improving treatments etc.).

[0047] However, it is clear to the average skilled person that the fibers as disclosed and claimed herein may comprise additives, such as inorganic fillers etc. in usual amounts as long as the presence of these additives has no detrimental effect on dope preparation and spinning operation. The type of such additives as well as the respective addition amounts are known to the skilled person.

Examples:

Example 1**:** Comparison of fibrillation dynamics

[0048] 2 different fiber types were produced using pulps with different hemicelluloses content (table 1). The fibers were prepared according to WO 93/19230, dissolving the pulps in NMMO and spinning over an air-gap into a precipitation bath to produce 1.3 dtex / 4 mm fibers (fiber 1 bright) according to the present invention; CLY standard bright (1.3 dtex / 4 mm) as reference standard lyocell fiber).

Table 1: Comparison of different hemicellulose contents of pulps

Sugar [%ATS]	Standard lyocell pulp	Hemicellulose-rich pulp
Glucan	95.5	82.2
Xylan	2.3	8.3
Mannan	0.2	5.7
Arabinan	<0.1	0.3
Rhaman	<0.1	<0.1
Galactan	<0.1	0.2

[0049] The 2 different fiber types were refined in an Andritz Laboratory plant 12-1C plate refiner (NFB, S01-218238) at a starting concentration of 6 g/l, 1400 rpm and 172 l/min flow rate. The gap was fixed at 1 mm.

[0050] The refining results are illustrated in figure 1. It can be seen that fiber 1 fibrillates at a significant higher rate compared to lyocell standard fibers, meaning a decrease in time- and energy effort.

Example 2: Fibrillation tendency

[0051] In table 2 the fibrillation properties evaluated according to CSF TAPPI Standard T227 om-94 after 8 minutes of mixing are reported. The fibers of the present invention show a vastly increased fibrillation tendency.

Table 2 : Comparison of CSF values of the fibers after 8 min of mixing time.

fiber type	CSF [ml]
1.3 dtex / 38 mmCLY standard bright	405
1.3 dtex / 38 mm fiber 1 bright	276

***Example 3*:** Pilling behavior

[0052] The lyocell fibers (1.3 dtex / 38 mm bright) produced according to example 1 were converted to Nm 50 ring yarns. These yarns were knitted on a Lawson&Hemphill FAK-S Sampler knitting machine, cylinder with 260 needles, 24 needles per inch, 54 gauge to produce knitted stockings. The samples were subjected to repeated washing cycles and evaluations of pill formation. In table 3 the results are reported, in relation with the pilling area as well as in relation with pilling count. The number of pillings was assessed using reference samples of EMPA Standard SN 198525 analogous to DIN EN ISO 12 945-2. This method was also published by Eldessouki et al (Fibers & Textiles in Eastern Europe 2014, 22, 6(108), 106-112).

Table 3: Comparison of pilling area and count.

	pill area after washing cycles [mm^2/dm^2]					pill count after washing cycles [$\#/dm^2$]				
Sample type	**0**	**1**	**5**	**10**	**15**	**0**	**1**	**5**	**10**	**15**
CLY standard bright	0	172	290	302	356	8	1508	2725	2775	3309
fiber 1 bright	0	35	140	41	62	0	417	1433	496	688

[0053] These results (shown in figure 2 and 3) prove the vastly improved properties of the fibers in accordance with the present invention. Pilling area as well as pilling count are decreased in comparison to standard lyocell fibers. Table 4 displays the respective results as % reduction of pill count and pill area. In combination with the increased fibrillation tendency as illustrated above, the fibers in accordance with the present invention do show a unique balance of properties, rendering the novel fibers valuable for a broad variety of applications.

Table 4: Reduction of pill area and count compared to standard lyocell fiber

Washing cycles	1	5	10	15
area	80%	52%	86%	83%
count	72%	50%	82%	80%

Claims

1. Lyocell fiber with decreased pill formation, having a hemicelluloses content of 5 wt.-% or more and a CSF value (8 min) of 350 ml or less.

2. Lyocell fiber according to claim 1, wherein the pill formation measured as the pill count, is at least reduced by 50% after 10 washing cycles compared to a standard Lyocell fiber with the same titer.

3. Lyocell fiber according to claim 1 or 2, wherein the CSF value is 300 ml or less.

4. Lyocell fiber according to any one of claims 1 to 3, wherein the hemicelluloses content is 10 wt.-% or more.

5. Lyocell fiber according to any one of claims 1 to 4, having a titer of 6.7 dtex or less, such as 2.2 dtex or less, preferably 1.3 dtex or less.

6. Lyocell fiber according to any one of claims 1 to 5, produced from a pulp having a hemicelluloses content of 7 wt.-% or more and 25 wt.-% or less and a xylan content of 6 wt.-% or more.

7. Lyocell fiber according to any one of claims 1 to 6, wherein the hemicellulose comprises a ratio of C5/xylan to C6/mannan of from 125:1 to 1:3, preferably of from 25:1 to 1:2.

8. Lyocell fiber according to any one of claims 6 or 7, wherein the pulp comprises 5 wt.-% or more xylan, preferably 8 wt.-% or more, more preferably 10 wt.-% or more and/or 3 wt.-% or more mannan, preferably 5 wt.-% or more mannan, and/or 1 wt.-% or less mannan.

9. Use of a pulp for producing a fiber according to any one of claims 1 to 8, wherein the pulp has a hemicelluloses content of 7 wt.-% or more and 25 wt.-% or less.

10. Use according to claim 9, wherein the hemicellulose comprises a ratio of C5/xylan to C6/mannan of from 125:1 to 1:3, preferably of from 25:1 to 1:2.

11. Use according to any one of claims 9 or 10, wherein the pulp comprises 5 wt.-% or more xylan, preferably 8 wt.-% or more, more preferably 10 wt.-% or more and/or 3 wt.-% or more mannan, preferably 5 wt.-% or more mannan, and/or 1 wt.-% or less mannan.

12. Method for producing the lyocell fiber according to any one of claims 1 to 8 using a direct dissolution process.

13. Method for producing the lyocell fiber according to claim 12 using a amine oxide process, where an aqueous solution of the amine oxide and the pulp form a cellulose suspension and a shapeable solution which gets shaped and coagulated in a spin bath obtaining the lyocell fiber after washing and pre-treatment steps.

14. Method for producing the lyocell fiber according to claim 13 using an aqueous tertiary amine oxide, preferably aqueous NMMO.

15. Method according to any one of claims 12 to 14, wherein the pulp comprises 10 wt.-% or more of hemicelluloses.

16. Product, comprising the lyocell fiber according to any one of claims 1 to 8, or the fiber produced according to any one of claims 12 to 15.

17. Product according to claim 16, wherein the product is a non-woven fabric.

18. Product according to claim 16 and/or 17, selected among tissues and wipes.

Figure 1

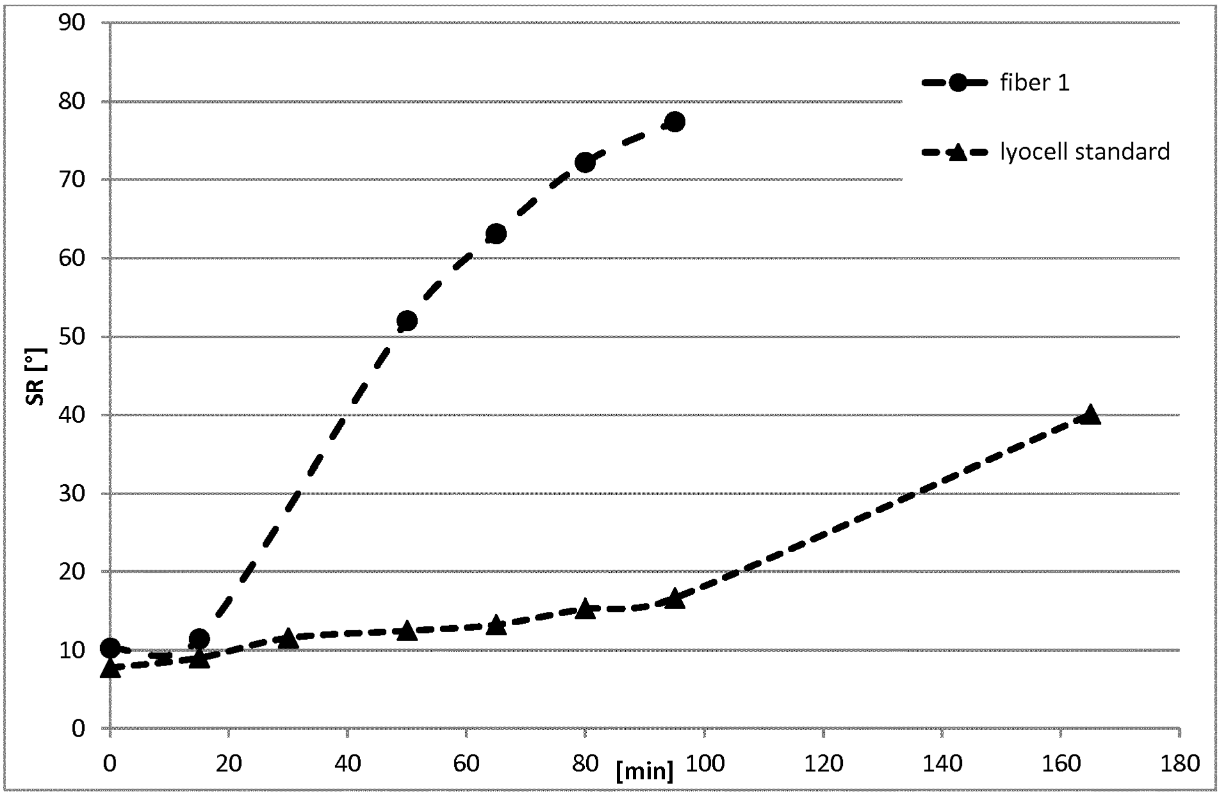

Figure 1: Fibrillation dynamics of two types of fibers

Figure 2

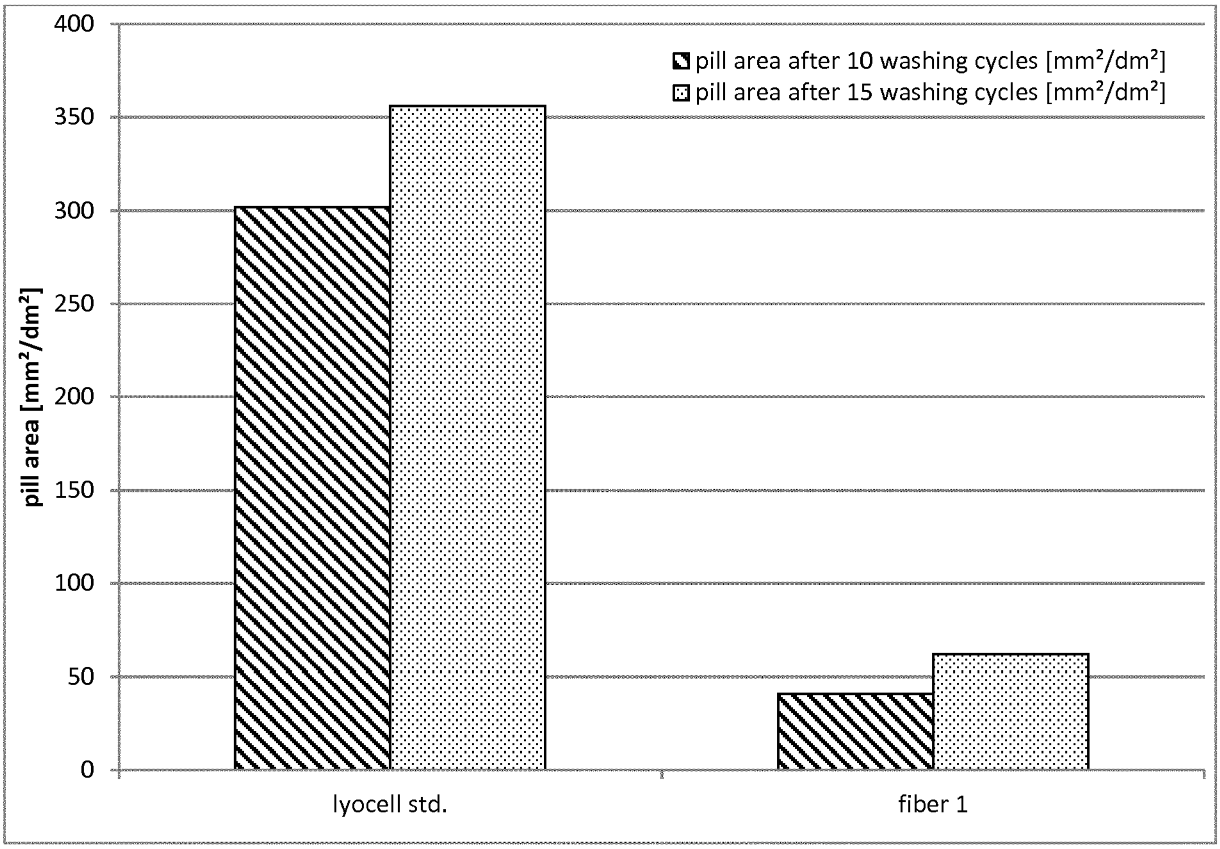

Figure 2: Pilling area after 10 and 15 wash cycles of two types of fibers

Figure 3

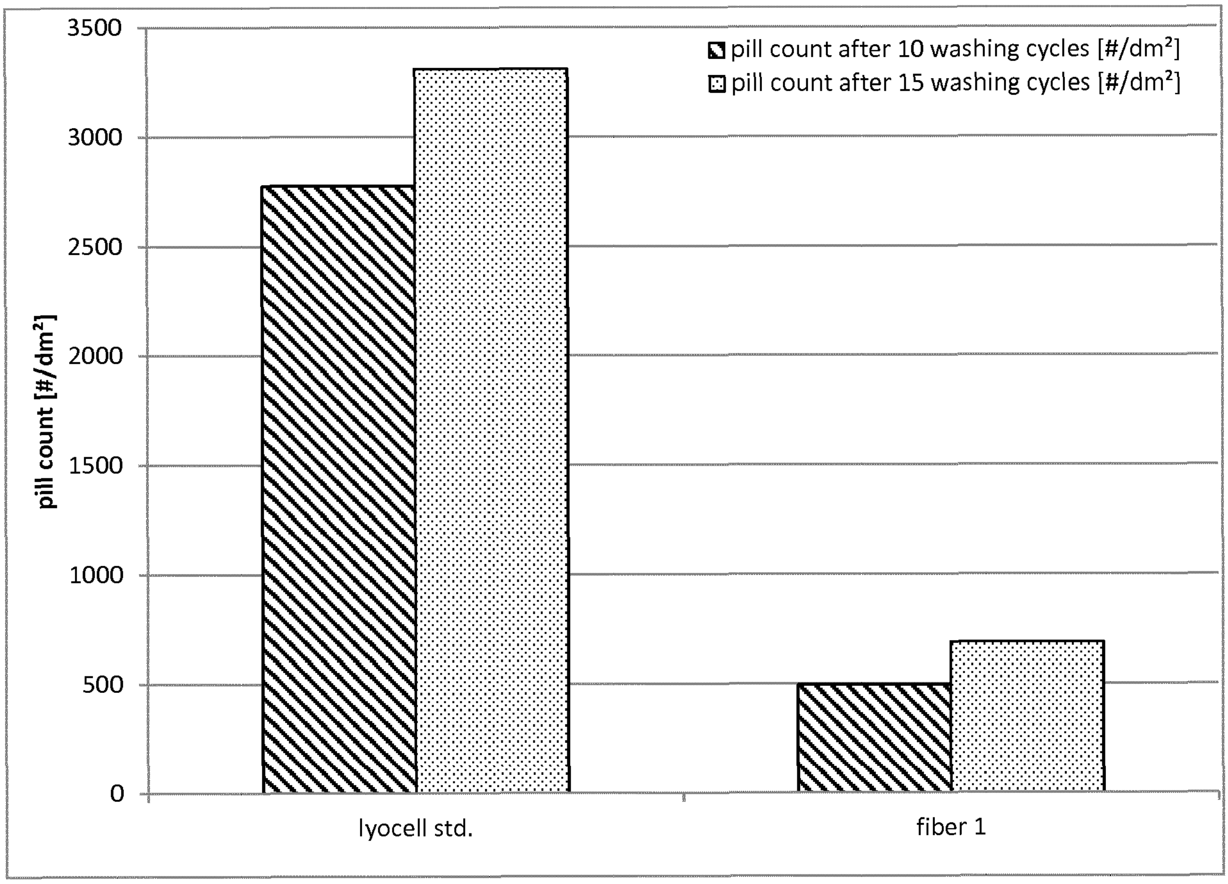

Figure 3: Pilling count after 10 and 15 wash cycles of two types of fibers

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EUROPEAN SEARCH REPORT

Application Number
EP 18 16 0143

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D21H
D01F

The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
Munich	23 April 2018	Ponsaud, Philippe

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