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(71) Applicant: **Lenzing Aktiengesellschaft**
4860 Lenzing (AT)

(72) Inventors:
- **ÖZTÜRK, Hale Bahar**
  4860 Lenzing (AT)
- **ULRICH, Julia**
  5303 Thalgau (AT)
- **MAIER, Ramona Anja**
  4863 Seewalchen (AT)

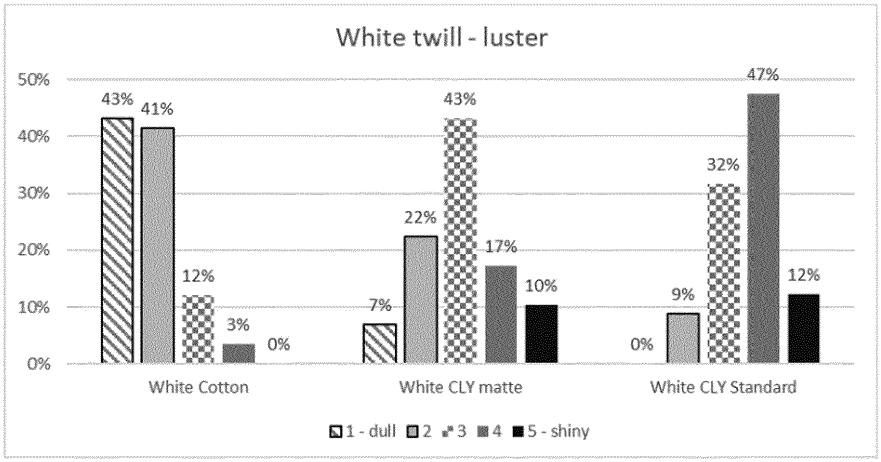
(74) Representative: **Schwarz & Partner Patentanwälte GmbH**
Patentanwälte
Wipplingerstraße 30
1010 Wien (AT)

(54) LYOCELL FIBRE COMPRISING A MATTING AGENT AND ITS USE FOR THE PRODUCTION OF A TEXTILE FABRIC

(57) The invention shows a lyocell fibre containing a matting agent whereby the matting agent comprises between 2 wt.-% and 5 wt.-% $BaSO_4$ and less than 1 wt.-% TiO_2, with respect to fibre weight, whereby the matting agent is incorporated in the fibre matrix in the form of particulates.

The invention further concerns the use of said fibre for the production of a textile fabric and a textile fabric containing said fibres.

Fig. 1a



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Description

[0001] The present invention relates to a lyocell fibre comprising a matting agent, the use of said lyocell fibre for the production of a textile fabric and a textile fabric comprising said lyocell fibre.

Prior art

[0002] Cotton is the most widely used natural cellulosic fibre for the production of textile fabrics. Although cotton is a very resistant fibre and can be subjected to aggressive and harsh treatments (e.g. denim laundry, etc.), its mechanical and haptic properties (e.g. handfeel, softness, etc.) are not always ideal in some applications.

[0003] Cellulosic man-made fibres can completely or partially replace cotton fibres in many applications. Suitable cellulosic man-made fibres in this regard include regenerated cellulose fibres such as viscose and modal or solvent-spun cellulosic fibres such as lyocell. Lyocell fibres are particularly suitable as a replacement for cotton due to their mechanical properties (such as high strength) and other properties (e.g. moisture management, grip, etc.). Especially in terms of handfeel and softness, lyocell fibres are regarded as superior to cotton. Lyocell fibres, however, have a high gloss which is undesirable for certain applications or for fashion reasons.

[0004] It is known from the prior art (WO 2010/144925 A1), that the gloss or sheen of lyocell fibres can be reduced by incorporating TiO_2 as a matting agent into the lyocell spinning mass. However, TiO_2 is very hard and abrasive, leading to high wear on the parts of the spinning apparatus. To achieve strong matting effects, a large amount of TiO_2 needs to be incorporated in the fibres, thus, increasing the wear and reducing the lifetime of the expensive equipment even further.

Disclosure of the invention

[0005] Therefore, the invention has the object of providing a lyocell fibre which has a similar appearance to cotton, while retaining its mechanical strength and other properties, such as handfeel, softness, etc., that can be produced cost-efficiently.

[0006] The invention achieves the stated object in that the matting agent may contain between 2 wt.- % and 5 wt.-% $BaSO_4$ and less than 1 wt.-% TiO_2, with respect to fibre weight, whereby the matting agent is incorporated in the fibre matrix in the form of particulates.

[0007] By incorporating 2 to 5 wt.-% $BaSO_4$ particulates in the fibre matrix, the disadvantages of the prior art can be overcome.

[0008] Furthermore, the present invention also solves the stated object by using the inventive fibre for the production of a textile fabric according to claim 6.

[0009] Even further, the present invention solves the stated object by providing a textile fabric according to claim 8 and a textile artic according to claim 14.

[0010] Preferred embodiments of the invention are set out in the dependent claims.

Brief description of the drawings

[0011] The embodiment variants of the invention are described in more detail below with reference to the drawings:

Fig. 1a shows the results of visual evaluation of the matte property of fabrics according to Example 1,
Fig. 1b shows the results of visual evaluation of the matte property of fabrics according to Example 2,
Fig. 2a shows a CIE LCh-Plot of results of visual evaluation of the matte property of fabrics according to Example 1 dyed with red reactive dye,
Fig. 2b shows a CIE LCh-Plot of results of visual evaluation of the matte property of fabrics according to Example 1 dyed with black reactive dye,
Fig. 2c shows a CIE LCh-Plot of results of visual evaluation of the matte property of fabrics according to Example 1 dyed with blue reactive dye,
Fig. 2d shows a CIE LCh-Plot of results of visual evaluation of the matte property of fabrics according to Example 1 directly dyed with blue dye,
Fig. 2e shows a CIE LCh-Plot of results of visual evaluation of the matte property of fabrics according to Example 1 directly dyed with green dye,
Fig. 2f shows a CIE LCh-Plot of results of visual evaluation of the matte property of fabrics according to Example 1 directly dyed with orange dye,
Fig. 2g shows a CIE LCh-Plot of results of visual evaluation of the matte property of fabrics according to Example 1 dyed with yellow VAT dye,
Fig. 2h shows a CIE LCh-Plot of results of visual evaluation of the matte property of fabrics according to Example 1

dyed with red VAT dye,

Fig. 3a shows the results of reflection measurements of fabrics according to Example 1 at 60° angle,

Fig. 3b shows the results of reflection measurements of fabrics according to Example 1 at 85° angle,

Fig. 3c shows the results of reflection measurements of fabrics according to Example 2 at 60° angle,

Fig. 3d shows the results of reflection measurements of fabrics according to Example 2 at 85° angle,

Fig. 4a shows an SEM image depicting the fibre surface of matted lyocell fibres according to the invention,

Fig. 4b shows an SEM image depicting the fibre surface of matted lyocell fibres according to the invention,

Fig. 4c shows an SEM image depicting the fibre cross-section of a single matted lyocell fibres according to the invention,

Fig. 4d shows an SEM image depicting the fibre cross-sections of multiple matted lyocell fibres according to the invention,

Fig. 4e shows an SEM image depicting the fibre surface of standard lyocell fibres,

Fig. 4f shows an SEM image depicting the fibre cross-sections of a cotton fibre, and

Fig. 5 shows a comparison of TSA spectra for undyed twill fabrics of matted lyocell fibres according to the invention and comparative standard lyocell fibres and cotton fibres.

Ways of implementing the invention

[0012] According to a first embodiment of the invention, the matting agent comprises between 2 wt.-% and 5 wt.-% $BaSO_4$ and less than 1 wt.-% TiO_2, whereby the matting agent is incorporated in the fibre matrix in the form of particulates. The wt.-%-values of $BaSO_4$ and TiO_2 are specified with respect to the fibre weight.

[0013] Due to the soft, non-abrasive mechanical properties of $BaSO_4$, the wear on all parts of the spinning device can be reduced, thus, increasing the service life of the spinning device. Furthermore, it has been found, that a combination of a low amount of TiO_2 (less than 1 wt.-%) and 2 to 5 wt.-% $BaSO_4$ leads to a strong matting effect in the fibres, which results in an appearance similar to cotton. Even after dyeing the fibres, the matting effect still remains, rendering the inventive fibres suitable for all kinds of textile applications, such as denim fabrics and the like. While the desired matting effect can be achieved by the inventive fibres, the softness and handfeel, known from textiles made from lyocell fibres, as well as the mechanical properties remain essentially unchanged. Thus, a lyocell fibre with matte appearance can be provided without sacrificing the mechanical fibre properties, which can also be produced cost-efficiently.

[0014] In a preferred embodiment of the invention, the matting agent comprises between 3 wt. % and 5 wt.-% $BaSO_4$ and between 0.5 wt.-% and 0.9 wt.-% TiO_2, with respect to the fibre weight. By reducing the amount of TiO_2 to less than 0.9 wt.-%, the abrasive effect on the spinning machine parts can be minimized, while providing a strong matting effect in combination with 3 to 5 wt.-% $BaSO_4$.

[0015] In an even further preferred embodiment, the matting agent comprises between 3.5 wt.-% and 4.5 wt.-% $BaSO_4$, more preferably between 3.8 wt.-% and 4.2 wt.-% $BaSO_4$, and between 0.6 wt.-% and 0.8 wt.-% TiO_2, with respect to the fibre weight.

[0016] In a further embodiment the $BaSO_4$ particulates may have a particle size distribution defined by an x_{50} lower than 1.5 μm and an x_{99} lower than 4 μm.

[0017] In an even further embodiment the TiO_2 particulates may have a particle size distribution defined by an x_{50} lower than 1 μm and an x_{99} lower than 2 μm.

[0018] In the context of the present invention, the x_{50}-value represents the mean particle size of the particle size distribution, while the x_{99}-value represents the particle size, where 99% of all particles are smaller than x_{99}.

[0019] The matting agent is added to the cellulose solution (i.e. the lyocell spinning mass) prior to spinning of the fibres. Therefore, the matting agent is preferably added to the spinning mass in the form of a suspension, where $BaSO_4$ and optionally TiO_2 particulates are dispersed in water beforehand with the help of a dispersion device. By supplying the particulates in the form of above mentioned particle size distributions, a very even distribution of the particulates in the spinning mass and subsequently in the fibres is possible.

[0020] It has been found, that adding $BaSO_4$ particulates as specified above to the spinning mass resulted in a uniform distribution of the particulates in the fibres, leading to lyocell fibres with strong mechanical properties (e.g. high tenacity). The particulates thereby have been found to be predominately arranged in the centre of the fibre core. A weakening of the fibre structure, increased fibrillation or abrasive properties on the fibre surface could not be observed. Thus, an improved matted lyocell fibre with high mechanical strength may be provided.

[0021] Advantageously the inventive lyocell fibres exhibit a titre from 1.1 dtex to 2.5 dtex, preferably from 1.3 dtex to 2.0 dtex.

[0022] Furthermore, the inventive lyocell fibres as described above may advantageously be used for the manufacture of a textile fabric.

[0023] According to one embodiment of the invention, the manufacture of the fabric comprises spinning a yarn using the lyocell fibre and dyeing at least a part of the yarn, more particularly with an indigo dye.

[0024] When dyeing lyocell fibres or fabrics made thereof, in particular with indigo dyes (indigo dyestuff), the natural shine of the fibres is further increased. Lyocell fibres have a natural shine due to the intrinsic fibre properties (e.g., smooth cross-section). The combination of lyocell fibres with brilliant colours (e.g., indigo, reactive, direct, or other dye-colours) bring about a shiny fabric surface. As indigo lyocell fabrics have more sheen than indigo cotton fabrics, a matte version of such lyocell denim fabrics is sought after in the fashion industry.

[0025] In the present invention, the term "denim" refers to a category of fabrics, which are not only characterized by the weave- or binding type but show a characteristic combination of properties. While for traditional denim fabrics a twill weave was used, other types of textile bindings (such as canvas or satin weave) have also become established in the modern textile industry. However, all denim fabrics are characterized by a robust woven fabric with dyed warp yarns and undyed weft yarns, whereby the warp is surface dyed, but the core of the warp remains undyed, which results in fading characteristics typical for denim. Due to the warp-faced weaving, denim is coloured on the outside and white (i.e., undyed) on the inside. Thereby, preferably indigo dyes are used for dyeing the warp.

[0026] A very common denim fabric is a woven fabric in which the warp contains indigo dyed cotton yarn while the weft contains undyed cotton yarn. Variations in denim fabric are however limitless e.g.: the weft yarn may contain dyed fibre (e.g., spun-dyed), the indigo dyed warp yarn can be overdyed with sulphur dyes, etc.

[0027] Alternative denim fabrics are also limitless, such as knit fabrics (e.g., circular, flat or seamless knits) containing indigo dyed cotton yarn. Alternative to cotton, other cellulosic yarns (such as lyocell, modal, viscose, etc.) can also be used for indigo dyeing. Denim fabrics have the ability to get wash-down/used look during garment laundries (ozone, laser, etc) thanks to indigo and other dischargeable dyestuffs.

[0028] By using the inventive fibre for spinning a yarn and subsequently dyeing at least part of the yarn, the advantages of the inventive fibre may be fully utilized. In particular, it has been found that the inventive fibres provide a matte appearance similar to cotton, even after dyeing. This is further demonstrated on behalf of several examples below.

[0029] In one embodiment of the invention, the first and second yarn may be interweaved to form a woven fabric, where the first yarn is a warp and the second yarn is a weft or vice-versa.

[0030] In particular, the woven fabric may be a twill fabric, a canvas fabric, a statin fabric or the like.

[0031] In a further embodiment, the woven fabric may be a 2/1 twill fabric with a basis weight between 100 g/m^2 and 300 g/m^2, preferably between 150 g/m^2 and 250 g/m^2, more preferably between 175 g/m^2 and 225 g/m^2. The warp having between 20 and 60 ends/cm, preferably between 30 and 50 ends/cm, more preferably between 32 and 48 ends/cm. The weft having between 10 and 35 picks/cm, preferably between 15 and 30 picks/cm, more preferably between 18 and 28 picks/cm. Such a twill fabric may show excellent opacity and matte properties in both undyed and dyed state (using a wide variety of different dyes). Further, such fabric shows a high smoothness and softness, comparable to standard lyocell fibres.

[0032] In a further embodiment, the second yarn also comprises lyocell fibres according to any of claims 1 to 5. In an alternative embodiment, the second yarn can also comprise cotton fibres or other cellulosic fibres, such as man-made cellulosic fibres, or synthetic fibres, such as polyester, etc.

[0033] In a further embodiment, the fabric has a wrinkle recovery rate of 60 % or greater.

[0034] In a further embodiment, the textile fabric has an opacity of 0.75 or greater, more preferably of 0.8 or greater.

[0035] In a preferred embodiment, the fabric is a denim fabric and the first yarn comprises an indigo dye on its surface. Due to the matted effect of the inventive fibres, denim fabrics made of said fibres have a solid matte effect, which can remain even after dyeing the fabrics with indigo dyestuff Thus, the inventive fibre is perfectly suited for the application in indigo textiles, such as denim.

[0036] In another preferred embodiment, the first and or second yarn are reactive dyed, direct dyed or vat dyed. Besides dyeing with indigo dyestuff, the advantages of the inventive fibres are also obtained when dyeing with reactive dyes, direct dyeing or dyeing with vat dyes.

[0037] Such fabrics as described above may advantageously used in textile articles, such as jeans, shirts, t-shirts, dresses, trousers, jackets, or the like.

Examples

[0038] In the following, the invention is further demonstrated on examples of textiles made from the inventive lyocell fibres.

[0039] For the evaluation of the fibre properties, the textile fabrics are compared with equivalent fabrics made from standard (unmatted) lyocell fibres and cotton fibres.

Example 1

[0040] In a first example a white undyed woven twill fabric was produced from a lyocell (CLY) fibre containing approx. 4 wt.-% $BaSO_4$ and approx. 0.75 wt.-% TiO_2 as matting agent according to the present invention (furthermore referred

to as "CLY Matte twill fabric").

[0041] The $BaSO_4$ particulates exhibited a particle size distribution characterized by: x_{10} = 0.42 μm, x_{16} = 0.56 μm, x_{50} = 1.09 μm, x_{84} = 1.78 μm, x_{90} = 2.05μm and x_{99} = 3.46 μm. The TiO_2 particulates exhibited a particle size distribution characterized by: x_{10} = 0.15 μm, x_{16} = 0.18 μm, x_{50} = 0.44 μm, x_{84} = 0.93 μm, x_{90} = 1.08μm and x_{99} = 1.49 μm.

[0042] The lyocell fibre thereby had a titre of 1.7 dtex and a staple length of 38 mm.

[0043] Fabric construction was a 2/1 twill with a basis weight of approx. 200 g/m2, warp: 38 ends/cm, weft: 20 picks/cm. Yarn properties were approx. Ne 20 for warp and Ne 24 for weft.

[0044] For comparison, a standard lyocell fibre with a titre of 1.3 dtex and a staple length of 38 mm was used to produce a 2/1 twill fabric with properties as defined above (referred to as "CLY Std. twill fabric").

[0045] Furthermore, for comparison, a cotton 2/1 twill fabric with properties as defined above was produced (referred to as "Cotton twill fabric").

Example 2

[0046] In a second example, a denim woven twill fabric was produced from a lyocell fibre containing the same amounts and characteristics of matting agents as specified in Example 1 (referred to as "CLY Matte denim fabric").

[0047] The lyocell fibre also had a titre of 1.7 dtex and a staple length of 38 mm.

[0048] Fabric construction was a 2/1 twill with a basis weight of approx. 200 g/m2, warp: 42 ends/cm, weft: 25 picks/cm. The warp has been indigo-dyed and the weft is left undyed.

[0049] Again, for comparison, a standard lyocell fibre with a titre of 1.3 dtex and a staple length of 38 mm and a cotton fibre were both used to produce denim fabrics (referred to as "CLY Std. denim fabric" and "Cotton denim fabric") with properties as defined above.

Matte Property - Visual Evaluation

[0050] First, the matte property of the fabrics of Examples 1 and 2 were evaluated visually by conducting an internal survey and, second, by analysing the CIELCh colour space (the CIELCh colour space is defined by the International Commission on Illumination).

[0051] In Fig. 1a the results of visual evaluation of fabrics according to Example 1 is shown. On the left, the survey of the white cotton twill fabric is shown; in the centre, the survey of the inventive white lyocell twill fabric with matting agent is shown and on the right, the survey of the standard lyocell twill fabric is shown.

[0052] The white cotton twill fabric is perceived by 43 % of people as dull (1) and by 41 % of people as more or less dull (2). 0 % of people perceived the cotton twill fabric as shiny (5).

[0053] The inventive matted lyocell twill fabric according to Example 1 is perceived by 43 % of people as neither dull nor shiny (3) and by 22 % of people as more or less dull (3). 7 % perceived it as dull (1), while 10 % perceived it as shiny (5).

[0054] The standard lyocell twill fabric is perceived by 47 % of people as more or less shiny (4) and by even 12 % as shiny (5). 0 % considered the standard lyocell fabric as dull (1).

[0055] This survey clearly shows that the inventive fibre produces a solid matting effect when used to manufacture a twill fabric. Although cotton is perceived as even more dull, a significant improvement over standard lyocell fabrics can be discerned.

[0056] In Fig. 1b the results of visual evaluation of fabrics according to Example 2 is shown. On the left, the survey of the cotton denim fabric is shown; in the centre, the survey of the inventive lyocell denim fabric with matting agent is shown; on the right, the survey of the standard lyocell denim fabric is shown.

[0057] Similar to what has been described above, a significant improvement in matting effect of the inventive fibre can be discerned from the survey results. In the denim fabrics according to Example 2, the effect is even more pronounced as for the white twill fabric according to Example 1. While for the standard lyocell denim fabric, the shine is increased due to the dyeing, the inventive matted lyocell fibre retains its dull or matte effect even after dyeing with indigo, making the appearance overall more similar to cotton.

Matte Property - CieLCh colour space

[0058] In the following, the matte properties of Example 1 and Example 2 fabrics are analysed by means of CieLCh colour space. All measurements are performed using a D65 light source under 10° angle.

[0059] The L*C*h colour space is preferred by some industry professionals because its system correlates well with how the human eye perceives colour. It has the same diagram as the L*a*b* colour space but uses cylindrical coordinates instead of rectangular coordinates.

[0060] In this colour space, L indicates lightness, C represents chroma, and h is the hue angle. The value of chroma C is the distance from the lightness axis L and starts at 0 in the centre. Hue angle starts at the +a axis and is expressed

in degrees (e.g., 0° is +a, or red, and 90° is +b, or yellow).

[0061] The L-axis describes the lightness (luminance) of the colour. Positive L means that product has lighter colour. Negative L means that product has darker colour.

[0062] The C-axis describes the chroma of a colour. Higher values mean brighter colour and lower value means duller colour.

[0063] The h-axis describes the wrinkle hue.

[0064] In Table 1, the CieLCh-Values for inventive lyocell matte, lyocell std. and cotton denim fabrics according to Example 2 are presented. The values are represented as Δ-values with respect to the lyocell std. values as a reference. From said Δ-values, changes in the parameters can be easily discerned.

Table 1: CieLCh-Values for Example 2 denim fabrics

Fabric type	CIE L	CIE a	CIE b	CIE C	CIE h	CIE Y
CLY Std. denim	26.91	0.55	-7.54	7.56	274.19	5.06
Cotton denim	23.63	0.39	-4.75	4.76	274.25	9.37
CLY Matte denim	29.19	-0.19	-7.03	7.03	273,46	7.52

[0065] From Table 1, it becomes obvious, that the lyocell matte denim fabric according to the present invention has less chroma (ΔC = -0.53), thus is duller, than the denim fabric from standard lyocell fibres.

[0066] For further evaluation, the white twill fabrics according to Example 1 were dyed with either red, black and blue reactive dyestuffs, blue, green and orange direct dyes or yellow and red vat dyes at same dyeing conditions but at separate dyebaths, so that fibre property was the only differing parameter. Table 2 shows CieLCh values of lyocell matte, lyocell standard and cotton fabrics, respectively for the different dyes. Higher L-values of inventive lyocell matte fabric for almost all dyes/colours than for lyocell standard fabric proves less lightness/shininess of matte lyocell compared to standard lyocell. Dyed cotton fabric was found to have least lightness/shininess compared to matte lyocell and standard lyocell.

[0067] Figures 2a-2c show the CIE LCh-Plots for reactive dyed textiles; Figures 2d-2f show the CIE LCh-Plots for direct dyed textiles; and Figures 2g-2h show the CIE LCh-Plots for VAT dyed textiles, respectively.

Table 2: CieLCh-Values for Example 1 twill fabrics dyed with different dyes (reactive dyes, direct dyes or VAT dyes)

Dye / Fabric type	CIE L	CIE a	CIE b	CIE C	CIE h	CIE Y
Red reactive dye						
CLY Std. twill	45.08	55.55	35.35	65.85	32.47	14.60
Cotton twill	48.66	55.02	31.33	63.33	29.30	20.00
CLY Matte twill	46.81	57.33	35.07	67.22	31.29	17.10
Black reactive dye						
CLY Std. twill	14.53	0.35	-0.41	0.54	310.62	1.82
Cotton twill	15.83	0.24	-1.51	1.53	310.13	3.52
CLY Matte twill	15.79	0.17	-1.47	1.48	310.10	3.47
Blue reactive dye						
CLY Std. twill	33.37	-0.66	-41.20	41.20	269.08	7.71
Cotton twill	37.25	-3.28	-41.25	41.38	266.47	12.39
CLY Matte twill	34.37	0.67	-42.25	42.25	267.67	9.68
Blue direct dye						
CLY Std. twill	53.45	-29.49	-33.25	44.44	228.43	21.46
Cotton twill	55.81	-29.75	-34.90	45.86	229.32	24.35
CLY Matte twill	53.04	-29.94	-33.08	44.60	227.99	22.09
Green direct dye						
CLY Std. twill	47.17	-44.10	17.31	47.37	158.57	16.15

(continued)

Green direct dye						
Cotton twill	49.16	-45.79	17.01	47.84	159.45	18.40
CLY Matte twill	46.26	-46.43	15.83	49.05	160.76	19.06
Orange direct dye						
CLY Std. twill	56.57	44.69	57.36	72.71	52.08	24.48
Cotton twill	58.48	46.03	60.97	76.39	53.21	28.78
CLY Matte twill	58.00	45.78	66.26	80.53	56.46	33.56
Yellow VAT dye						
CLY Std. twill	81.38	4.65	87.48	87.61	86.96	59.16
Cotton twill	83.55	1.95	88.03	88.06	89.68	62.67
CLY Matte twill	83.09	0.88	87.80	87.81	90.74	63.31
Red VAT dye						
CLY Std. twill	37.84	45.87	7.63	46.50	9.44	10.00
Cotton twill	38.11	46.12	6.57	46.59	8.36	11.12
CLY Matte twill	40.28	47.68	2.60	47.75	4.25	15.87

Reflection Measurements

[0068] Figures 3a-3d show the determination of the gloss value of fabrics according to Examples 1 and 2 at 60° and 85°, respectively. The gloss value was determined according to DIN EN ISO 2813:2015-02 by means of a reflectometer REFO 3-D of Dr. Lange Company.

[0069] The test was carried out on the sample on the right side of the goods (marked by the weaver). 10 measurements per angle setting were carried out on the sample material on a non-reflective background (black spectrometer paper). The measurements were made longitudinally, diagonally and transversely to the warp/weft direction of the material. The sample was air-conditioned at 20 °C, 65 % relative humidity for at least 24 h prior to the measurements

[0070] The higher the value given in Figures 3a-3d, the stronger the shine.

[0071] As can be clearly seen from Figs. 3a and 3b, white twill fabrics according to Example 1 made of the inventive lyocell matte fibres and from cotton fibres show very similar reflection values, whereas values for fabrics made of lyocell standard fibres are significantly higher, hence they have a stronger shine.

[0072] A similar observation can be made for denim fabrics according to Example 2 as depicted in Figs. 3c and 3d. In all cases, reflection values of fabrics made of the inventive lyocell matte fibres and of cotton fibres are lower than for fabrics made of lyocell standard fibres, which again indicates a stronger shine.

Opacity property of the invention

[0073] Furthermore, the opacity of fabrics using the lyocell fibre containing matting agents according to the present invention is analysed.

[0074] Opacity in the context of the present invention refers to the opacity or opaqueness of the investigated textiles. The opacity is indicated as a unitless number between 0 and 1 (or 0% and 100%), whereby perfect transparency is present at 0, which decreases with increasing value. At 1, the respective material is completely opaque.

[0075] In all measurements, opacity has been determined at a wavelength of 570 nm with a Konica Minolta CM600d spectrophotometer (Q425F168, Inv. No. 71559) including accessory set for calibration. Enclosed computer (laptop) with SpectramagicNX software was used for evaluation. Measurements have been calibrated using TQC-Test (Chart Art. No. VF2345 Batch No. 227270) and Green tile CM-A101GN.

[0076] Opactiy values were determined with the above setup following NWSP060.4.R0.20 (defined for measuring opacity of nonwovens).

[0077] In Table 3, the measured opacity-values for undyed twill fabrics according to Example 1, as described above, are shown. Therein it can be discerned, that the twill fabric made from the inventive lyocell matte fibres has the highest opacity, similar to the twill fabric made from cotton. Both have a significantly higher opacity than the fabric made from

lyocell std. fibres.

Table 3: Opacity-values for undyed twill fabrics according to Example 1

Fabric type	Opacity
CLY Std. twill	0.74 ± 0.01
Cotton twill	0.81 ± 0.01
CLY Matte twill	0.83 ± 0.01

[0078] Furthermore, in Table 4, the measured opacity-values for single knit jersey fabrics (Ne 20/1 ring spun, 110 gsm) made from the lyocell matte fibre according to the invention and from lyocell standard fibre are shown, whereby the fabrics have been dyed with different dyes.

[0079] Dyeing of the fabrics was performed at different dye baths using the same recipes so that there is no difference between fibres regarding dye uptake.

[0080] As can be discerned from Table 4, coloured jersey knit fabrics from lyocell matte fibres show consistently higher opacity values than similar jersey knit fabrics made from lyocell standard fibres.

Table 4: Opacity-values for dyed jersey knit fabrics

Dye / Fabric type	Opacity
Yellow color (Novacron Yellow FN-2R)	
CLY Std. knit	0.58 ± 0.01
CLY Matte knit	0.72 ± 0.01
Orange color (Novacron Orange FN-R)	
CLY Std. knit	0.76 ± 0.01
CLY Matte knit	0.82 ± 0.01
Red color (Novacron Brilliant red FN-3GL)	
CLY Std. knit	0.83 ± 0.01
CLY Matte knit	0.86 ± 0.01
Blue color (Novacron Brilliant Blue FN-G)	
CLY Std. knit	0.81 ± 0.01
CLY Matte knit	0.90 ± 0.01
Blue Color (Novacron Marine F-G)	
CLY Std. knit	0.76 ± 0.01
CLY Matte knit	0.89 ± 0.01

Fibre smoothness

[0081] In Figs. 4a - 4d, SEM (scanning electron microscopy) images of the surface (Fig. 4a, 4b) and cross-section (Fig. 4c, 4d) of the lyocell matte fibres according to the present invention are depicted.

[0082] In Fig. 4e an SEM image of the surface of lyocell standard fibres is shown, and in Fig. 4e, the cross-section of a cotton fibre is depicted.

[0083] The SEM images clearly show that the fibre surface is smooth without any visible defects caused by the incorporation of matting agents. This can also be discerned from the cross-section images, which show a smooth regular shape and cross-section without visible defects, as would be expected from lyocell fibres. Thus, the matting agent is finely dispersed in the fibre matrix and does not form agglomerations or the like.

Mechanical properties (strength, elongation)

[0084] Further, tensile strength and elongation of the lyocell matte fibres according to the invention and comparative

lyocell standard fibres were tested according to the BISFA standard ("Testing methods for viscose staple fibers).

[0085] Strength- and elongation values of the fibres are shown in Table 5. Thereby it can be discerned, that lyocell matte fibres have fibre strength and elongation similar to lyocell standard fibres without matting agents. Only a 6 % decrease in fibre strength can be observed.

Table 5: Fibre strength and elongation values

Fibre type	Fibre strength (cN/tex)	Fibre elongation (%)
CLY Std. fibre	34.8 ± 0.5	12.7 ± 0.5
CLY Matte fibre	32.6 ± 0.6	11.0 ± 0.5

[0086] Furthermore, the strength and elongation of yarns made from lyocell matte fibres according to the present invention and comparative lyocell standard and cotton fibres is evaluated. Yarn configurations for all fibres were: Ne 20 (Nm 34, 300 dtex), 661T/m. Strength- and elongation-values are given in Table 6, respectively.

Table 6: Yarn strength and elongation values

Yarn type	Yarn strength (cN/tex)	Yarn elongation (%)
CLY Std. yarn	28.1 ± 0.5	6.9 ± 0.5
Cotton yarn	15.8 ± 0.5	6.3 ± 0.5
CLY Matte yarn	16.4 ± 0.5	10.0 ± 0.5

[0087] From the values it can be clearly discerned, that yarn strength and elongation of the lyocell fibres containing matting agents still remain high and exceed that of cotton.*Fabric Softness*

[0088] Softness and surface properties (roughness/smoothness) of undyed woven twill fabrics (Ne 20/1, 110 gsm) using the lyocell matte fibre (CLY Matte) according to the present invention and comparative fabric using standard lyocell fibre (CLY Std.) and cotton fibre were measured by a Tissue Softness Analyzer (TSA type B0458) device manufactured by Emtec Electronic (Germany). The analysis of fabrics by use of TSA is well known to the skilled person (see e.g.: Abu-Rous et al., J Fashion Technol Textile Eng 2018, S4).

[0089] The rotating part of the TSA generates noise while moving over the fabric surface, which is captured by a microphone and analysed into its amplitude signals. In the resulting sonic spectrum, the signal peak (in dB V^2 rms) at 750 Hz (TS750) is a measure for the fabric vibration under the rotating part and should correlate with fabric smoothness, while the peak at 6500 Hz (TS7) occurs through the vibration on the rotating part itself while moving above the fabric surface and is considered a measure for the softness of surface fibres. The lower the generated noise, the smoother resp. softer is the fabric (higher TS750 peak means higher roughness, lower TS7 peak means higher softness).

[0090] Fig. 5 shows measured TSA spectra of the undyed woven twill fabrics for Cotton, CLY Std. and inventive CLY Matte fibres. The results extracted from the spectra are also summarised in Table 8. Higher TS750 and TS7 peaks correspond to lower smoothness and lower softness, respectively. From both Fig. 5 and Table 8 it can be clearly discerned, that the twill fabrics made of matte lyocell fibres according to the present invention show very similar (high) smoothness and softness as standard lyocell fibres without matting agent. Twill fabrics made from cotton fibres on the other hand show increased roughness and reduced softness.

[0091] Thus, it is demonstrated, that the inventive lyocell fibres comprising the matting agent according to the claims can maintain the high softness and smoothness as usually expected for lyocell fibres.

Table 8: Tissue Softness Analyser Test (TSA) results for undyed woven twill fabrics

Fabric type	TS7	TS750	D (mm/N)
CLY Std. twill	13.325 ± 0.4	32.592 ± 2.9	1.76 ± 0.03
Cotton twill	18.915 ± 1.6	73.543 ± 6.4	1.41 ± 0.05
CLY Matte twill	11.651 ± 1.2	34.874 ± 2.5	1.42 ± 0.02

Claims

1. Lyocell fibre comprising a matting agent, **characterized in that** the matting agent comprises between 2 wt.-% and

5 wt.-% $BaSO_4$ and less than 1 wt.-% TiO_2, with respect to fibre weight, whereby the matting agent is incorporated in the fibre matrix in the form of particulates.

2. Lyocell fibre according to claim 1, **characterized in that** the matting agent comprises between 3 wt.-% and 5 wt.-% $BaSO_4$ and between 0.5 wt.-% and 0.9 wt.-% TiO_2, with respect to fibre weight.

3. Lyocell fibre according to claim 1 or 2, **characterized in that** the $BaSO_4$ particulates having a particle size distribution defined by an x_{50} lower than 1,5 μm and an x_{99} lower than 4 μm.

4. Lyocell fibre according to any of claims 1 to 3, **characterized in that** the TiO_2 particulates having a particle size distribution defined by an x_{50} lower than 1 μm and an x_{99} lower than 2 μm.

5. Lyocell fibre according to any of claims 1 to 4, **characterized in that** the fibre exhibits a titre from 1.1 dtex to 2.5 dtex, preferably from 1.3 dtex to 2.0 dtex.

6. Use of a lyocell fibre according to any of claims 1 to 5 for the manufacture of a textile fabric.

7. Use according to claim 6, **characterized in that** the manufacture of the fabric comprises spinning a yarn using the lyocell fibre and dyeing at least a part of the yarn, more particularly with an indigo dye.

8. Textile fabric, having at least a first yarn comprising lyocell fibres according to any of claims 1 to 5 and a second yarn, whereby the first and second yarn are interweaved to form the textile fabric.

9. Textile fabric according to claim 8, **characterized in that** the second yarn comprises lyocell fibres according to any of claims 1 to 5.

10. Textile fabric according to claim 8 or 9, **characterized in that** the fabric has a wrinkle recovery rate of 60 % or greater.

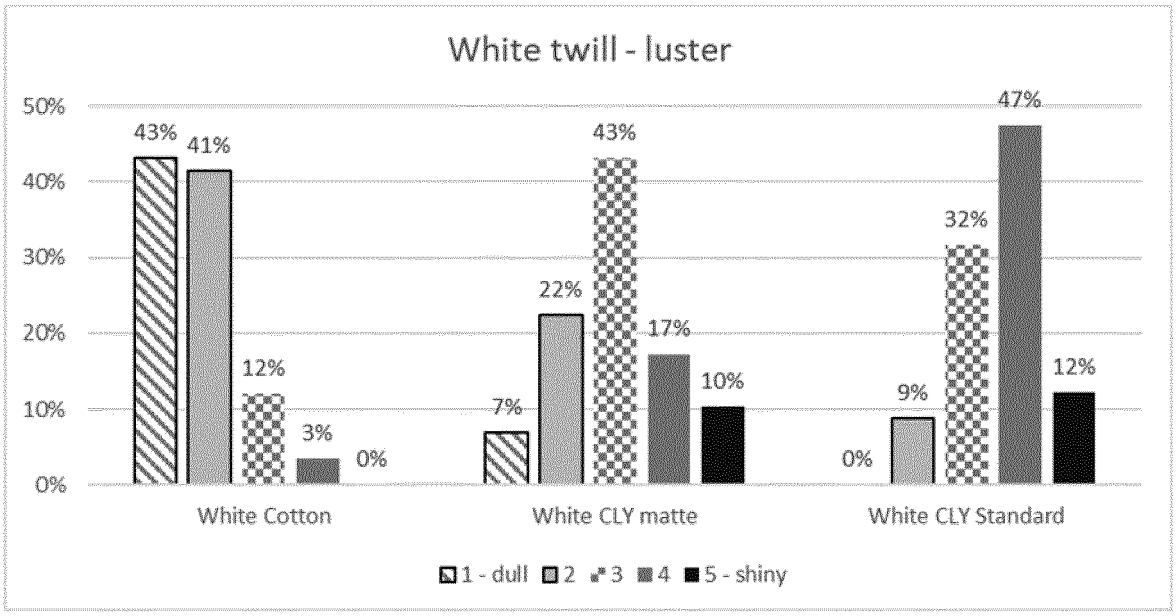
11. Textile fabric according to any of claims 8 to 10, **characterized in that** the fabric has an opacity of 0.75 or greater, more preferably of 0.8 or greater.

12. Textile fabric according to any of claims 8 to 11, **characterized in that** the fabric is a denim fabric and the first yarn comprises an indigo dye on its surface.

13. Textile fabric according to any of claims 8 to 11, **characterized in that** the first and or second yarn are reactive dyed, direct dyed or vat dyed.

14. Textile article comprising a textile fabric according to any of claims 8 to 12.

Fig. 1a


White twill - luster
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40%
30%
20%
10%
0%
43%
41%
12%
3%
0%
7%
22%
43%
17%
10%
0%
9%
32%
47%
12%
White Cotton
White CLY matte
White CLY Standard
1 - dull
2
3
4
5 - shiny

Fig. 1b

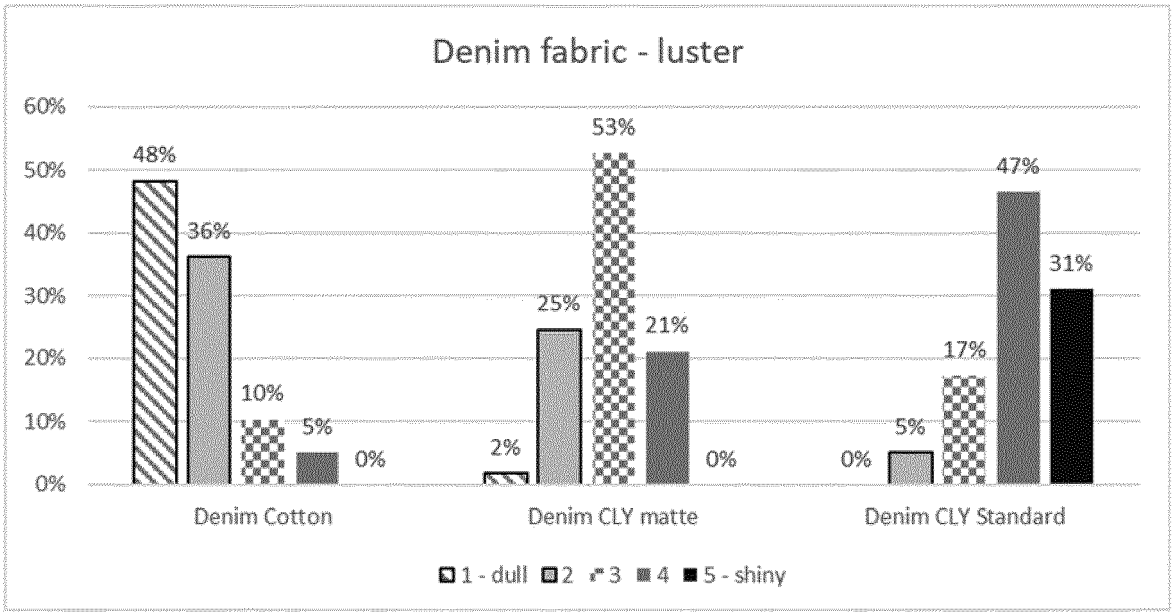

Denim fabric - luster
60%
50%
40%
30%
20%
10%
0%
48%
36%
10%
5%
0%
2%
25%
53%
21%
0%
0%
5%
17%
47%
31%
Denim Cotton
Denim CLY matte
Denim CLY Standard
1 - dull
2
3
4
5 - shiny

Fig. 2a

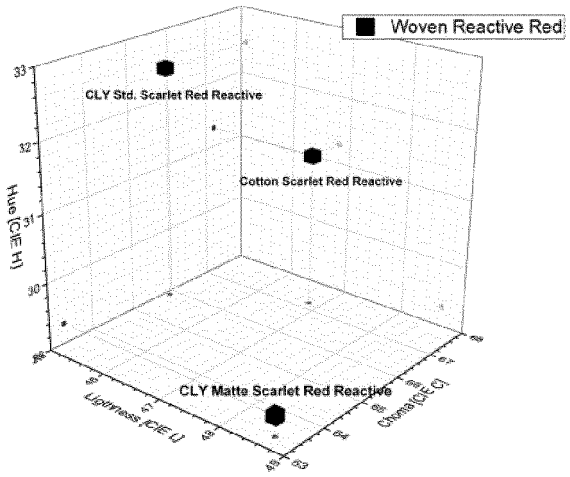


Fig. 2b

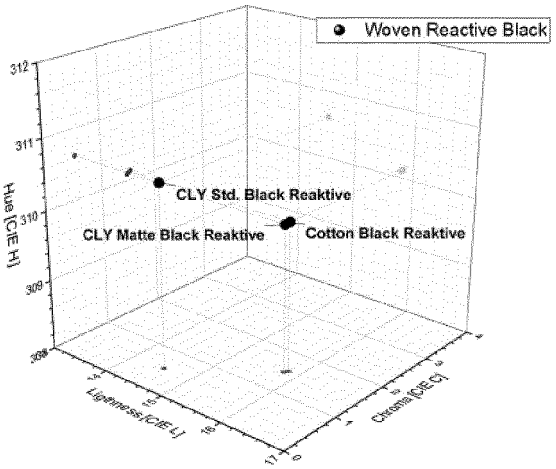


Fig. 2c

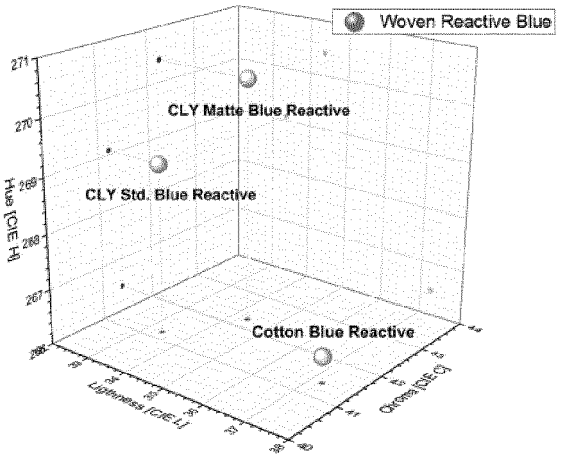


Fig. 2d

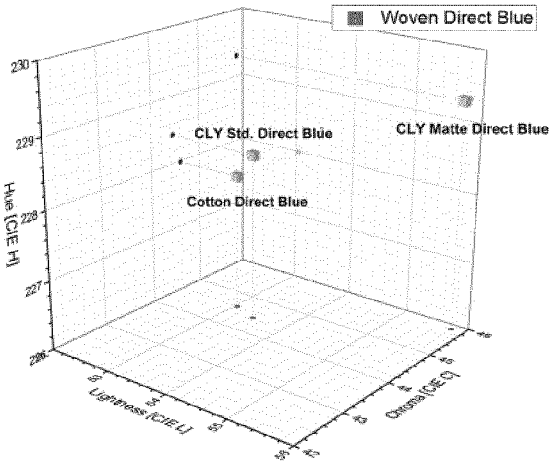


Fig. 2e

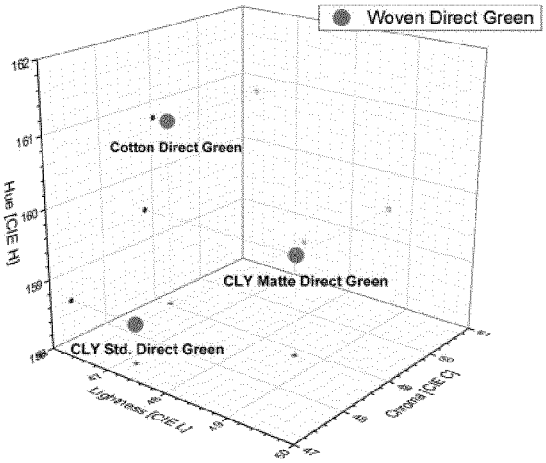


Fig. 2f

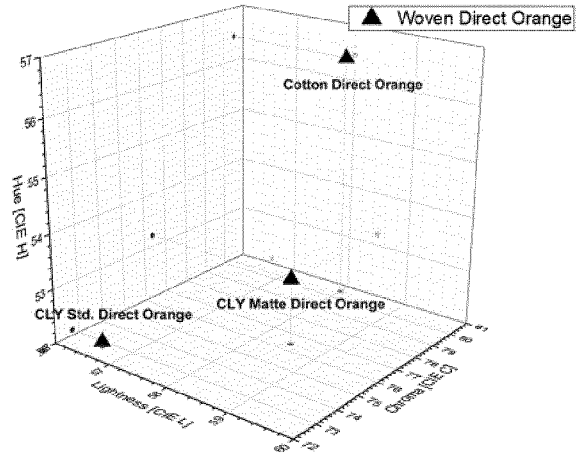
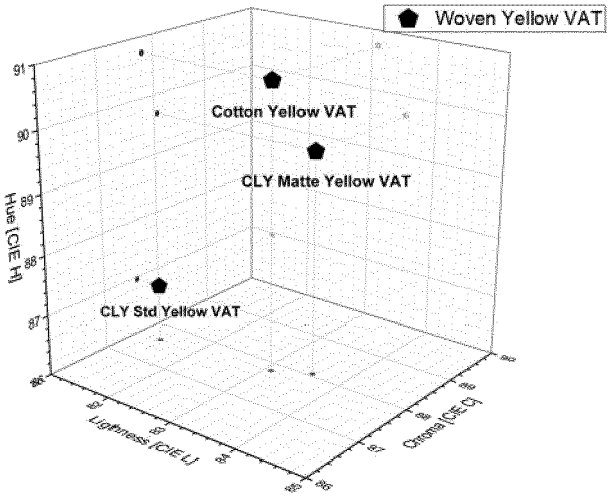


Fig. 2g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CLY Matte Yellow VAT
CLY Std Yellow VAT
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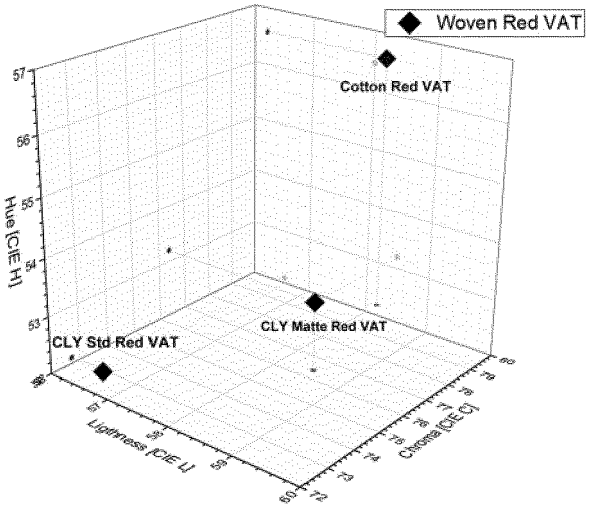
Lightness [CIE L]
Chroma [CIE C]

Fig. 2h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CLY Matte Red VAT
CLY Std Red VAT
Hue [CIE H]
Lightness [CIE L]
Chroma [CIE C]

Fig. 3a

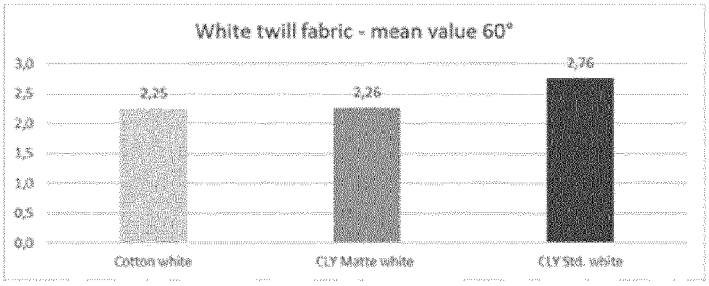


Fig. 3b

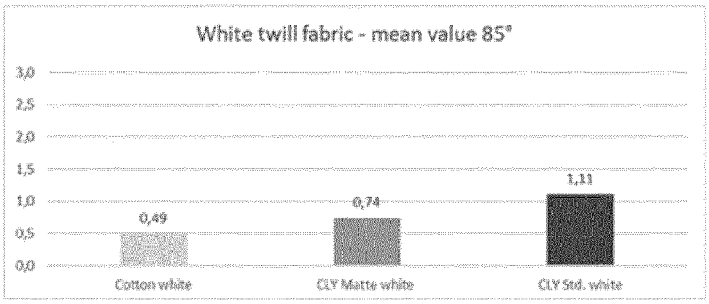


Fig. 3c

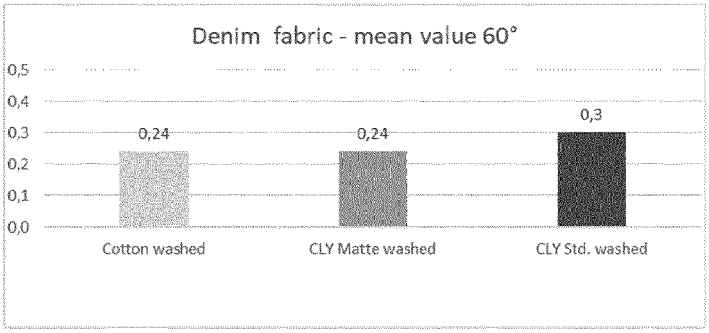


Fig. 3d

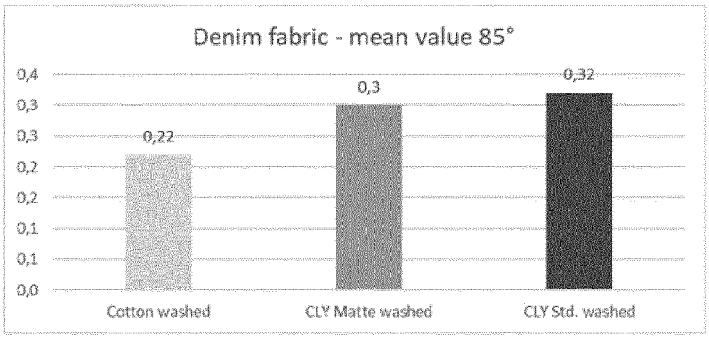


Fig. 4a

Fig. 4b

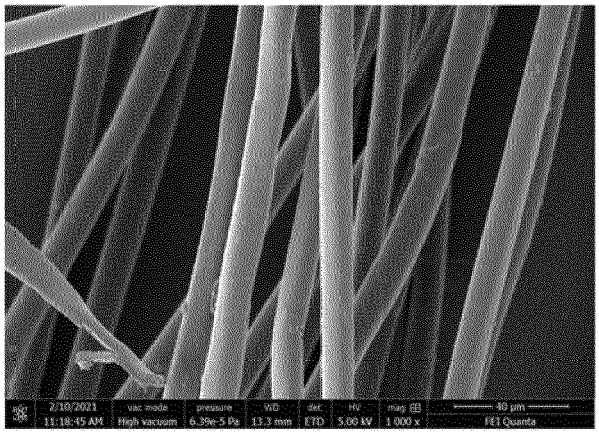


Fig. 4c

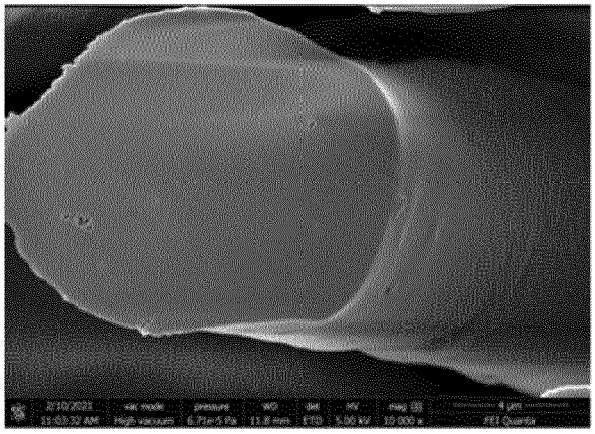


Fig. 4d

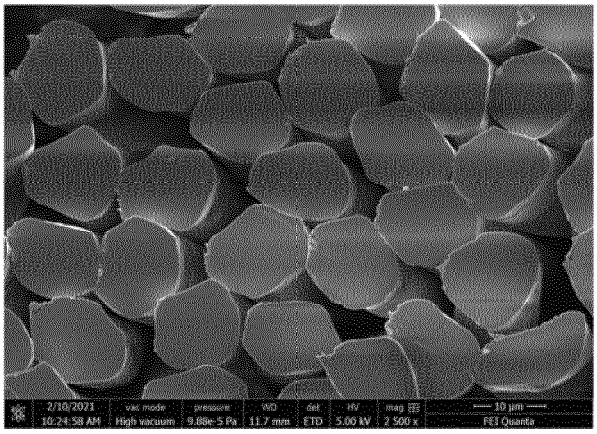


Fig. 4e

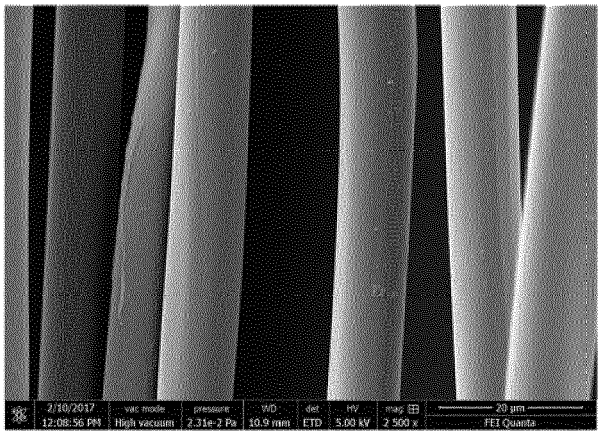


Fig. 4f

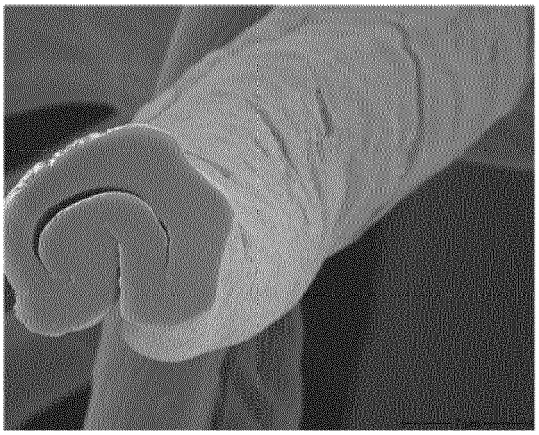
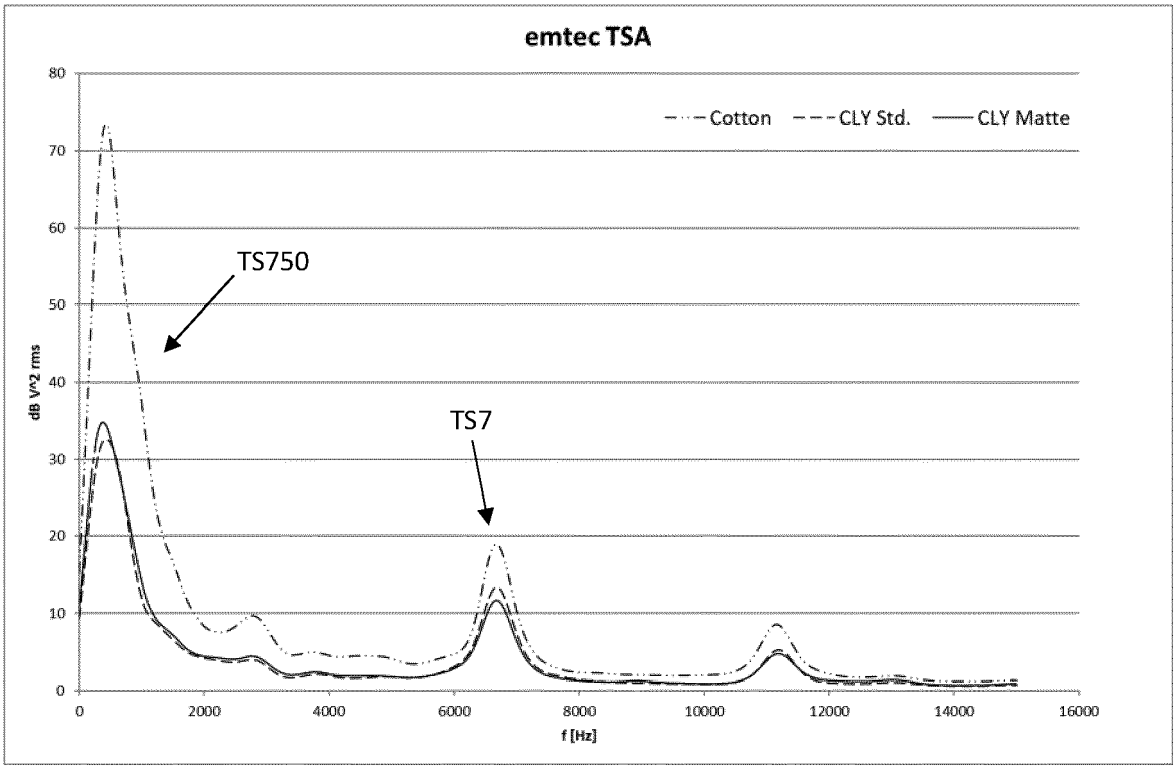


Fig. 5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Cotton
CLY Std.
CLY Matte
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TS7
dB V^2 rms
f [Hz]
0
10
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30
40
50
60
70
80
0
2000
4000
6000
8000
10000
12000
14000
16000

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EUROPEAN SEARCH REPORT

Application Number
EP 21 19 9580

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	WO 2019/170670 A1 (CHEMIEFASER LENZING AG [AT]) 12 September 2019 (2019-09-12) * claims 1,3,4,5 *	1-14	INV. D01F2/00 D01F1/10 D01F1/06
A	WO 2019/068476 A1 (CHEMIEFASER LENZING AG [AT]) 11 April 2019 (2019-04-11) * abstract; claims 1-14 *	1-14	D06P1/22 D06P3/66 D03D15/225 D03D15/54
A	EP 3 511 371 A1 (CHEMIEFASER LENZING AG [AT]) 17 July 2019 (2019-07-17) * abstract; claims 1-6 *	1-14	
A	US 5 895 795 A (HASHEMZADEH ABDULMAJID [DE]) 20 April 1999 (1999-04-20) * column 4, lines 52-61; claims 1-6,15-26; examples 1,2 *	1-14	

TECHNICAL FIELDS SEARCHED (IPC)
D01F
D06Q
D06P
D03D

1 The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	7 March 2022	Malik, Jan

CATEGORY OF CITED DOCUMENTS

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07-03-2022

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 2019170670 A1	12-09-2019	BR 112020016978 A2	15-12-2020
		CA 3091720 A1	12-09-2019
		CL 2020002131 A1	15-01-2021
		CN 111819314 A	23-10-2020
		EP 3762525 A1	13-01-2021
		JP 2021517213 A	15-07-2021
		KR 20200130273 A	18-11-2020
		TW 201940767 A	16-10-2019
		US 2020407883 A1	31-12-2020
		WO 2019170670 A1	12-09-2019
WO 2019068476 A1	11-04-2019	AU 2018345581 A1	12-03-2020
		BR 112020004280 A2	29-09-2020
		CN 111183249 A	19-05-2020
		EP 3467171 A1	10-04-2019
		EP 3692196 A1	12-08-2020
		JP 6989079 B2	05-01-2022
		JP 2020536185 A	10-12-2020
		KR 20200057764 A	26-05-2020
		PE 20201036 A1	09-10-2020
		RU 2753288 C1	12-08-2021
		TW 201923185 A	16-06-2019
		US 2020291551 A1	17-09-2020
		WO 2019068476 A1	11-04-2019
EP 3511371 A1	17-07-2019	CN 111655771 A	11-09-2020
		EP 3511371 A1	17-07-2019
		JP 2021511447 A	06-05-2021
		TW 201940522 A	16-10-2019
		WO 2019138092 A1	18-07-2019
US 5895795 A	20-04-1999	AU 4941696 A	23-09-1996
		CA 2214402 A1	12-09-1996
		CN 1177364 A	25-03-1998
		EP 0813573 A1	29-12-1997
		JP H11501071 A	26-01-1999
		KR 19980701923 A	25-06-1998
		PL 322109 A1	05-01-1998
		SK 115397 A3	04-02-1998
		TW 442525 B	23-06-2001
		US 5895795 A	20-04-1999
		WO 9627638 A1	12-09-1996

EPO FORM P0459

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Patent documents cited in the description

- WO 2010144925 A1 **[0004]**

Non-patent literature cited in the description

- **ABU-ROUS et al.** *J Fashion Technol Textile Eng,* 2018, S4 **[0088]**