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(54) **Improvement of specular gloss of viscose fiber**

(57) Improvement of specular gloss of viscose fiber using nanocrystalline cellulose

The invention relates to a process for imparting gloss onto the surface of a viscose fiber material, characterized in that
(v) a viscose fiber material is immersed in a liquid hydrophilic medium to give a swollen viscose fiber material,
(vi) said swollen viscose fiber material is treated with a suspension or solution of nanocrystalline cellulose in a liquid hydrophilic medium,
(vii) said treated viscose fiber material is washed with water and optionally with aqueous sulfuric acid,
(viii) said washed viscose fiber material is dried.

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Description

[0001] The invention relates to imparting, preferably enhancing the gloss of viscose fibers using nanocrystalline cellulose, hereinafter called NCC. Viscose is regenerated cellulose, insoluble in water, prepared by treatment with sodium hydroxide and carbon disulfide and made water soluble to form sodium cellulose-xanthate solution. The slurry when passed through a spinneret under pressure into sulfuric acid solution bath yields regenerated cellulose material known as viscose. In principle, the chemistry of cellulose and viscose remains the same with exception of physical properties such as crystallinity and morphology. Regeneration of cellulose is a precipitation process carried out in harsh acidic condition which affects significantly physical parameters such as crystallinity of the viscose fiber material.

[0002] Specular gloss is an ability of a surface to reflect light in the specular direction in relation to that of a polished flat glass surface with a well-defined optical property such as refractive index. Gloss is identified as a surface phenomenon or effect and flat surfaces empower gloss effect.

[0003] In principle, the industrial process of viscose fiber manufacturing can be divided into two parts, namely the pre-regeneration and the post-regeneration regime by means of which improvements in effective specular gloss value can be achieved. Multiple additives in sodium cellulose-xanthate slurry reduce the refractive index of regenerated cellulose and lower the effective gloss. Slurries with pigment dispersions are sometimes not spinnable due to harsh acidic regeneration (cellulose to viscose) conditions. In post-regeneration regime of cellulose, finishing chemicals such as special oils are used to impart gloss or finish to the fiber. The use of additives and finish chemicals can impart gloss to the finished fiber to a certain extent. Gloss obtained by such treatment can be attributed to adsorbed finishing chemicals such as surfactants or special oils used.

[0004] Viscose fibers lack the crystallinity on fiber contour regions. Precipitation conditions such as temperature and molecular weight of cellulose affects the crystallinity of the viscose fiber.

[0005] The present invention helps to overcome the deficiency in gloss on fiber surface. The invention also provides a novel concept of the ability of nano-crystalline cellulose (NCC) to alter physical properties of fiber surfaces such as viscose fiber material leading to an enhancement of specular gloss.

[0006] The present invention relates to a process for imparting gloss onto the surface of a viscose fiber material, characterized in that

(i) a viscose fiber material is immersed in a liquid hydrophilic medium to give a swollen viscose fiber material,
(ii) said swollen viscose fiber material is treated with a suspension or solution of nanocrystalline cellulose in a liquid hydrophilic medium,
(iii) said treated viscose fiber material is washed with water and optionally with aqueous sulfuric acid,
(iv) said washed viscose fiber material is dried.

[0007] The viscose fiber material used in the present invention can be any kind of regenerated cellulose, e.g. neat viscose or a blend of viscose with other fiber material, such as polyester or any natural or synthetic fibers.

[0008] It is also possible to use a colored viscose fiber material, e,g. colored with a dye or a pigment as specified hereinafter.

[0009] In step (i) the viscose fiber material is immersed in a liquid hydrophilic medium for a time sufficient to affect a swelling of the viscose fiber material. The liquid hydrophilic medium is advantageously an aqueous medium, an alcoholic medium or an aqueous alcoholic medium. The alcoholic medium comprises monovalent or polyvalent alcohols, preferably those which are soluble in water, e.g. methanol, ethanol, iso-propanol, n-propanol, n-butanol, iso-butanol, tert-butanol, glycols, such as ethylene glycol, propylene glycol, or glycerine. The aqueous medium can be water alone or comprise water in combination with water soluble organic solvents. The aqueous alcoholic medium comprises water or said aqueous medium and one or more of the alcohols mentioned before. Ethylene glycol is the preferred alcohol.

[0010] The swelling time according to step (i) is expediently at least 10 minutes, preferably 10 to 120 min, more preferably 20 to 90 min, most preferably 30 to 60 min. A swelling time of more than 120 min is also possible although usually of no technical advantage. For swelling an elevated temperature is advantageous, e.g. a temperature between 40 and 130 °C, preferably between 50 and 120 °C, more preferably between 60 and 110 °C, even more preferably between 70 and 100 °C.

[0011] Step (ii): Nanocrystalline cellulose (NCC) can be obtained from native fibers, e.g. wood fibers, by an acidic hydrolysis commonly using sulfuric or hydrochloric acid giving rise to nanoparticles or nanowhiskers which are rod like in shape with an aspect ratio which preferably varies from 1 to 100 depending on the cellulose source. Cellulose nanocrystals average 100 to 500 nm, preferably 150 to 300 nm, more preferably 180 to 200 nm, in length with a cross section of 1 to 20 nm, preferably 2 to 10 nm, more preferably 3 to 5 nm. NCC suspensions are stabilized in aqueous media by means of sulfate ester groups imparted to the cellulose nanocrystal surfaces. Sulfuric acid is used for said acidic hydrolysis of cellulose regeneration. The extent of suspension ability of NCC in aqueous or liquid hydrophilic medium may vary depending on the source and molecular weight of NCC.

[0012] The concentration of NCC in the liquid hydrophilic medium may range from 0.5 to 15 % strength by weight, preferably from 1 to 10 % strength by weight, more preferably from 2 to 7.5 strength % by weight, based on the total weight of the liquid hydrophilic medium and NCC.

[0013] The liquid hydrophilic medium in steps (i) and (ii) can be the same or different. For reasons of process economy, the liquid hydrophilic medium is the same in step (i) and (ii). It is possible that the whole or a partial amount of the NCC to be used is already present in the swelling step (i).

[0014] The amount by weight of NCC relative to the weight of the viscose fiber material to be treated may range from (0.10 to 10.0) : 1, preferably from (0.20 to 2.0) : 1, more preferably from (0.25 to 1.0) : 1, most preferably from (0.30 to 0.80) : 1.

[0015] The treatment time of the viscose fiber material with the aqueous suspension or solution of NCC is expediently at least 10 minutes, and may range from 10 to 120 min, preferably from 20 to 90 min, more preferably from 30 to 60 min. A treatment time of more than 120 min is also possible although usually of no technical advantage.

[0016] The temperature applied during said treatment may range from 40 to 130 °C, preferably from 50 to 120 °C, more preferably from 60 to 110 °C.

[0017] In step (iii) the treated viscose fiber material is washed with water, preferably several times and preferably with distilled water and preferably with water at a temperature of 20 to 40 °C. Optionally a further treatment with diluted sulfuric acid, e.g. with a 1 to 5 % strength by weight sulfuric acid, is carried out at a temperature of between 20 and 40 °C, preferably while ultrasonifying, followed by washing to neutral with water.

[0018] The drying step (iv) is preferably performed at a temperature of between 45 and 75 °C for preferably 2 to 8 hours.

[0019] The present invention further relates to a viscose fiber material wherein the viscose fibers are coated with nanocrystalline cellulose.

[0020] The present invention further relates to a viscose fiber material wherein the viscose fibers are coated with nanocrystalline cellulose by a process as specified above.

[0021] Said viscose fiber material can be any kind of regenerated cellulose, e.g. neat viscose or a blend of viscose with other fiber material, such as polyester or any natural or synthetic fibers.

[0022] The viscose fiber material can be colorless or can be a colored viscose fiber material. The colorant is selected from the group consisting of natural colorants derived from plants or animals and synthetic colorants, preferred synthetic colorants being synthetic organic and inorganic dyes and pigments, preferred synthetic organic pigments being azo or disazo pigments, laked azo or disazo pigments or polycyclic pigments, particularly preferably phthalocyanine, diketopyrrolopyrrole, quinacridone, perylene, dioxazine, anthraquinone, thioindigo, diaryl or quinophthalone pigments.

[0023] The thickness of the neat viscose fibers can be customary, preferably ranging from 5 to 50 μm, more preferably from 10 to 40 μm, even more preferably from 15 to 30 μm.

[0024] The thickness of the NCC coating layer on the viscose fiber is preferably in the range of from 0.5 to 25 μm, more preferably 1.0 to 20 μm, even more preferably 1.5 to 15 μm, most preferably 2.0 to 10 μm, especially 2.5 to 8 μm.

[0025] The coated viscose fiber material of the present invention is characterized by a gloss that is enhanced compared to the gloss of the respective uncoated viscose fiber material. Preferred are gloss value enhancements of at least 5 %, preferably at least 10 %, more preferably at least 15 %, measured with a glossmeter at an angle of 60°.

Examples:

Materials:

[0026] NCC material used in the following examples was sourced from CelluForce, Canada. Chemically the material is 100 % cellulose hydrogen sulphate sodium salt. It is easily soluble in water in a concentration of 1 to 2 % by weight. For concentrations above 3 % by weight, the solubility in water was improved by providing ultrasonic energy to the dispersion which results in a semi-translucent dispersion.

[0027] Colored viscose fiber (blue) with 150 denier was obtained from Century rayon Industries Mumbai. Copper-phthalocyanin blue was the colorant used to color the viscose fiber.

Examples 1 - 5:

[0028] A blue colored viscose fiber was wound on plastic plates and the so prepared swatches (weight: 20 g) were processed with an aqueous NCC solution or dispersion in water of different concentrations (Samples I to IV) or without NCC solution (Sample Blank, as comparative) in a Zeltex® Dyeing Machine.

[0029] The swatches were first kept in a 100 ml metallic bomb reactor containing distilled water or NCC solution (concentrations as of Samples I - IV) and then immersed in an ethylene glycol bath of 20 Liter for 30 - 45 min at 90 - 120 °C. One of these swatches served as blank and as an internal standard for evaluation. The swatches (Samples I - IV) were then treated at 90 - 110 °C for 30 to 45 min with varying concentrations of aq. suspensions or solutions of NCC of volume 100 ml (cf. Table 1).

[0030] The samples I - IV were then washed once with distilled water at room temperature (24 - 26 °C) followed by a 2 % strength by weight sulfuric acid solution treatment in an ultrasonic bath at room temperature for 10 - 15 min. The acid treatment was followed by repeated washings with water at room temperature.

[0031] The samples were dried at 40 - 60 °C in a drying oven for 4 - 6 hours.

[0032] The dried samples were measured for the gloss using a Minigloss meter supplied by M/s SDL International Textile testing solution. The angle of measurement was 60° and the temperature of the measurements was 24 - 26 °C. The results are given in Table 1 below.

Table 1: Gloss measurements

Sample Name	Concentration of NCC solution in wt.%	Gloss
Blank	0	6.3
Sample - I	1.0	6.82
Sample - II	2.0	7.0
Sample - III	3.0	7.2
Sample - IV	5.0	7.9

[0033] Treated and untreated samples were also studied using Scanning Electron Microscopy (SEM). SEM was used to assess the deposition of NCC on viscose fiber surface. Images showing the fiber width confirm the occurrence of coating of NCC on viscose fiber surface. The results are given in Table 2 below:

Table 2:

Sample Name	Width in micrometer after treatment
BLANK (no treatment)	24-25
Sample - I	28-30
Sample - II	30-33
Sample - III	34-37
Sample - IV	37-40

Claims

1. A process for imparting gloss onto the surface of a viscose fiber material, **characterized in that**

(i) a viscose fiber material is immersed in a liquid hydrophilic medium to give a swollen viscose fiber material,
(ii) said swollen viscose fiber material is treated with a suspension or solution of nanocrystalline cellulose in a liquid hydrophilic medium,
(iii) said treated viscose fiber material is washed with water and optionally with aqueous sulfuric acid,
(iv) said washed viscose fiber material is dried.

2. The process of claim 1, wherein the liquid hydrophilic medium is an aqueous medium, an alcoholic medium or an aqueous alcoholic medium.

3. The process as claimed in claim 1 or 2, wherein step (i) is carried out at a temperature of between 40 and 130 °C.

4. The process as claimed in any of the preceding claims, wherein the immersion in step (i) is carried out for at least 10 minutes.

5. The process as claimed in any of the preceding claims, wherein the nanocrystalline cellulose is present in the liquid hydrophilic medium in a concentration of from 0.5 to 15 % strength by weight.

6. The process as claimed in any of the preceding claims, wherein step (ii) is carried out at a temperature of between

40 and 130 °C.

7. The process as claimed in any of the preceding claims, wherein the treatment in step (ii) is carried out for at least 10 minutes.

8. The process as claimed in any of the preceding claims, wherein in step (iii) the aqueous sulfuric acid has a concentration of 1 to 5 % strength by weight.

9. The process as claimed in any of the preceding claims, wherein in step (iii) ultrasonic energy is applied during washing with aqueous sulfuric acid.

10. A viscose fiber material wherein the viscose fibers are coated with nanocrystalline cellulose.

11. The viscose fiber material as claimed in claim 10, wherein the viscose fibers are coated with nanocrystalline cellulose in a thickness of the coating layer on the viscose fiber in the range of from 0.5 to 25 μm.

12. The viscose fiber material as claimed in claim 10 or 11, wherein the viscose fibers are coated with nanocrystalline cellulose in a thickness of the coating layer on the viscose fiber in the range of from 1.5 to 15 μm.

13. The viscose fiber material as claimed in any of claims 10 to 12, wherein the viscose fibers are coated with nanocrystalline cellulose in a thickness of the coating layer on the viscose fiber in the range of from 2.0 to 8 μm.

14. The viscose fiber material as claimed in any of claims 10 to 13, wherein the viscose fibers are colored.

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EUROPEAN SEARCH REPORT

Application Number
EP 15 00 0175

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
The Hague	22 July 2015	Fiocco, Marco

CATEGORY OF CITED DOCUMENTS

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Application Number
EP 15 00 0175

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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The present search report has been drawn up for all claims

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