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(54) **PROCESS FOR DYEING AND FINISHING OF CELLULOSIC FABRICS**

VERFAHREN ZUM FÄRBEIN UND VEREDELN VON CELLULOSISCHEN GEWEBEN

PROCEDE POUR LA TEINTURE ET L'APPRET DES ETOFFES EN CELLULOSE

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Description**Field of the invention**

5 **[0001]** This invention relates to the dyeing and finishing of lyocell and also polynosic fabrics.

[0002] Lyocell fibres are known. Lyocell is the generic name for shaped cellulosic articles made by dissolution of cellulose in a solvent without formation of a chemical derivative of cellulose, followed by extrusion of the resulting solution into a coagulating bath to form a shaped cellulosic article. Suitable known solvents include a variety of tertiary amine N-oxides, often in admixture with water. One such tertiary amine N-oxide is N-methylmorpholine N-oxide. Polynosic fibres are also known. Polynosic is the generic name for regenerated cellulose fibres having a defined set of properties, made by the viscose process.

[0003] As used herein, a lyocell yarn is a yarn which contains or consists of lyocell fibre, and a lyocell fabric is a fabric which contains or consists of lyocell yarn. Polynosic yarn and polynosic fabric are to be construed similarly.

15 **[0004]** It is known that lyocell fibre has a tendency to fibrillate. The same is true of polynosic fibre but to a lesser degree. Fibrillation is a phenomenon which may occur when lyocell fibre is subjected to mechanical stress in the wet state, notably during the fabric processing steps typically involved in dyeing and finishing operations. The result of fibrillation is the partial detachment of fine fibrils from the body of the fibre, which can lead to a change in visual appearance and handle. Considerable efforts have been made to reduce or control the fibrillation tendency of lyocell fibre, in particular by the use of suitably-chosen manufacturing conditions or by the application of cross-linking agents.

20 **[0005]** Two types of fibrillation in lyocell fabric have been distinguished. Primary fibrillation involves the formation of long fibrils, often 1 mm or longer and often unevenly distributed over the fabric. In severe cases, so-called white-line defects may be seen, where extensive fibrillation has occurred at creases in the fabric. Primary fibrillation confers an unsightly appearance on lyocell fabrics and is generally considered to be undesirable. Secondary fibrillation is the formation of short fibrils, a fraction of a millimetre in length, uniformly and densely distributed over the fabric. In contrast to primary fibrillation, secondary fibrillation can confer an attractive appearance and handle on a fabric, often aptly called a peach-touch finish.

Background art

30 **[0006]** It is known that a preferred route towards the attainment of a peach-touch finish involves: fibrillation (or pre-fibrillation) by subjecting wet fabric to mechanical stress, for example during processing in rope form, thereby inducing primary fibrillation; defibrillation, i.e. removal of primary fibrils, for example by treatment with an enzymatic preparation containing a cellulase; and further fibrillation (or refibrillation) by subjecting the fabric to controlled mechanical stress in the wet state, thereby inducing secondary fibrillation and thus providing a peach-touch finish. Such a process is described, for example, in a paper entitled "Die Veredlung von Lyocellfasern - Bin Erfahrungsbericht" by R Breier in Lenzinger Berichte 9/94 at pages 99-101. Processing in rope form has the advantage that it provides bulking and relaxation of the fabric, leading to superior handle.

35 **[0007]** A conventional scheme for dyeing and finishing lyocell fabrics so as to provide a peach-touch finish involves the following steps in sequence. Scour the fabric, using an alkaline detergent solution, to remove sizes and other unwanted substances. Rinse with hot and cold water. Dye the fabric with a reactive dyestuff, if required in the presence of alkali (catalyst). Rinse the fabric several times with hot and cold water to remove unreacted dyestuff and to adjust fabric pH towards neutrality. (A typical rinsing sequence involves washing cold; washing at 70°C; washing at 40°C; boiling in detergent solution; washing at 70°C; washing at 40°C; and washing with cold water (seven steps).) (If some or all of the preceding steps are performed on fabric in rope form, the mechanical stresses involved serve to induce primary fibrillation.) Treat the fabric with an aqueous solution of a cellulase to remove primary fibrils. (It has previously been observed that the best results are obtained by the use of cellulases which are most active at acidic pH.) Heat the solution to denature the cellulase. Rinse with water. Induce secondary fibrillation by mechanical treatment, for example in a jet-processing machine, typically in water at 95°C for 45 minutes. Apply a softener to the fabric, typically from a water bath at 40°C for 30 minutes. Dry the fabric and then tumble-beat to perfect the peach-touch finish. In a typical optimised industrial process, such a scheme occupies 12-14 hours and requires the use of 12 liquor baths (both exclusive of the scouring step and its associated rinses).

50 **[0008]** It is an object of the invention to provide a method for dyeing and finishing lyocell and also polynosic fabrics which occupies less time and requires fewer liquor baths than such known schemes. Further objects and advantages of the invention appear hereinafter.

Disclosure of invention

55 **[0009]** According to the invention, there is provided a method for dyeing and finishing a lyocell fabric or a polynosic

fabric including the steps of:

(1) applying to the fabric in rope form a liquor containing in solution a reactive dyestuff for cellulose, under conditions such that both (i) a high degree of exhaustion of the dyestuff onto the fabric is achieved and (ii) fibrillation of the fabric occurs, thereby providing a dyed fabric exhibiting primary fibrillation;

(2) applying to the fabric in rope form with no more than a single intermediate rinse a solution of a cellulase, and maintaining the solution in contact with the fabric under conditions effective to remove primary fibrils therefrom by the action of the cellulase, thereby providing a dyed fabric exhibiting a low degree of fibrillation;

(3) raising the temperature of the solution whilst it is maintained in contact with the fabric, whereby both (i) the cellulase is denatured and (ii) secondary fibrillation is induced in the fabric, thereby providing a dyed fabric exhibiting secondary fibrillation; and

(4) drying the fabric.

[0010] The fabric is often a woven fabric, although knitted fabrics may also be used.

[0011] We have surprisingly found that the cellulase treatment is remarkably effective in removing unfixed dyestuff, without the need for the multiple rinsing steps required in conventional techniques.

[0012] The fabric may be prepared for dyeing in conventional manner. This typically involves scouring in open width with an alkaline detergent solution in order to remove sizes and suchlike materials from the fabric.

[0013] Equipment for the wet-processing of fabrics in rope form is known, for example jet-processing machines. Suitable equipment for use in the method of the invention includes Thies Luft-Roto (Trade Mark), Then AFS (Trade Mark) and Krantz Aerodye (Trade Mark) machines.

[0014] If desired, the liquor used in dyeing step (1) may contain a salt such as sodium sulphate in known manner and as recommended by dyestuff manufacturers to assist the dyeing process. If desired, the liquor used in dyeing step (1) may contain conventional fabric softening agents and/or lubricants.

[0015] The intermediate rinse which can precede step (2) is preferably omitted except in the case of deep and dark shades, obtainable by dyeing under alkaline conditions as more particularly described hereinafter.

[0016] The denaturing and refibrillation step (3) is conveniently performed by heating the liquor to a temperature in the range from 80 to 100°C and maintaining the liquor in contact with the fabric for sufficient time to achieve the specified effects.

[0017] Preferably, the denaturing and refibrillation step (3) is succeeded by a rinsing step (3a) to remove salts, proteinaceous matter and fibrillar debris from the fabric. The rinsing step (3a) may conveniently be performed using cold water.

[0018] Preferably, a softening step in which a fabric softener is applied to the fabric is carried out immediately prior to the drying step (4). In one embodiment of the invention, a conventional fabric softener is added to the bath containing the denatured cellulase at the end of the denaturing and refibrillation step (3), and the fabric is subsequently dried without intermediate rinsing. In another embodiment of the invention, the softener is added to the rinsing water in the optional rinsing step (3a). In a further embodiment of the invention, the softener is padded onto the fabric after the optional rinsing step (3a).

[0019] The drying step (4) may be performed in conventional manner. In suitable cases, it may conveniently be performed in the same equipment as used for the preceding wet-processing steps. In other cases, it may conveniently be performed on a tumbling machine. Continued tumbling of the fabric after drying is complete serves to lift the pile of the fabric and to remove therefrom loose fibres remaining from the defibrillation step, and this is a preferred additional step in the method of the invention.

[0020] Many reactive dyestuffs for cellulose react most rapidly with cellulose under alkaline conditions. Examples of such dyestuffs include those in the Procion H-E and Procion H-EXL (Trade Marks of BASF AG), Remazol (Trade Mark of DyStar) and Sumifix Supra (Trade Mark of Sumitomo Chemical Co. Ltd.) series. Many such dyestuffs are based on vinylsulphones or halotriazines. In such a case, the dyeing step (1) may be performed using an alkaline liquor, in accordance with the recommendations of the dyestuff manufacturer. We have nevertheless found that in the method of the invention an advantageous and novel embodiment of dyeing step (1) involves the following steps in sequence:

(i) applying to the fabric a liquor containing in solution a reactive dyestuff for cellulose, the solution containing no added alkali;

(ii) maintaining the liquor in contact with the fabric at a temperature in the range from 100 to 150°C until fibrillation has occurred;

(iii) cooling the liquor to a temperature within the range from 40 to 100°C;

(iv) adding alkali to the liquor; and

(v) maintaining the liquor in contact with the fabric in order to complete fixation of the dyestuff onto the fabric.

[0021] The pH of the dye liquor in step (i) may be controlled by addition of a pH buffer. A pH in the range from 5 to 8, often from 6 to 7, will be found suitable in many cases.

[0022] The amount of alkali added in step (iv) is preferably chosen in accordance with the recommendations of the dyestuff manufacturer.

[0023] The recommended dyeing temperature for many reactive dyestuffs is below 100°C. We have found that dyeing at a high temperature in the range from 100 to 150°C as in step (ii) has the advantages that primary fibrillation is more rapid and the handle of the fabric is softer. Late addition of alkali in step (iv) to the cooled liquor has the advantage that alkaline hydrolysis of the dyestuff is minimised, thus increasing fixation efficiency.

[0024] As mentioned above, it has previously been observed that the best results are generally obtained when using cellulases which are most active at acidic pH. Accordingly, when the exhausted liquor at the end of step (1) is alkaline, a preferred technique is to discard this liquor at the end of step (1) and to use in step (2) a solution of cellulase which contains a suitable pH buffering agent.

[0025] Reactive dyestuffs capable of reacting with cellulose in the absence of added alkali are also known. Examples of such dyestuffs include those made by reaction of a halotriazine with a tertiary amine to form a dyestuff containing a quaternary ammonium group. An example of a suitable tertiary amine is nicotinic acid. Dyestuffs of this kind are available commercially under the names Kayacelon React (Trade Mark of Nippon Kayaku Co. Ltd.) and Procion H-EG (Trade Mark of BASF AG). Other reactive dyestuffs capable of reacting with cellulose in the absence of added alkali are also known, for example some dyestuffs containing a halopolyazaine nucleus as reactive centre. Such dyestuffs may be utilised in similar manner to those which require alkaline conditions, as described hereinabove. Use of such dyestuffs nevertheless permits a further simplification of the method of the invention, as will now be described.

[0026] According to the invention, there is further provided a method for dyeing and finishing a lyocell fabric or a polynosic fabric including the steps of:

(A) applying to the fabric in rope form a liquor containing a reactive dyestuff in solution, the dyestuff being capable of reacting with cellulose in the absence of added alkali, under conditions such that both (i) a high degree of exhaustion of the dyestuff onto the fabric is achieved and (ii) fibrillation of the fabric occurs, thereby providing a dyed fabric exhibiting primary fibrillation;

(B) adding a cellulase to the liquor;

(C) maintaining the liquor in contact with the fabric under conditions effective to remove primary fibrils therefrom by the action of the cellulase, thereby providing a dyed fabric exhibiting a low degree of fibrillation;

(D) raising the temperature of the liquor whilst it is maintained in contact with the fabric, whereby both (i) the cellulase is denatured and (ii) secondary fibrillation is induced in the fabric, thereby providing a dyed fabric exhibiting secondary fibrillation; and

(E) drying the fabric.

[0027] The dyeing step (A) is preferably performed at a temperature in the range from 100 to 150°C. The pH of the dye liquor is typically slightly acid, for example in the range from 5 to 7, preferably 6 to 7, and accordingly contains no added alkali. The pH of the dye liquor may be controlled by addition of a suitable pH buffer. Dyeing at high temperature, for example at about 130°C, has the advantages that the dyeing reaction is rapid, primary fibrillation is more rapid and the handle of the dyed fabric is softer.

[0028] Step (B) involves addition of a cellulase to the exhausted dyeing liquor from step (A). The cellulase is conveniently supplied and added in the form of a liquid cellulase preparation. It will be understood that the liquor should if necessary be cooled to a sufficiently low temperature to avoid denaturing the enzyme upon addition to the liquor. If required, the pH of the liquor can be adjusted prior to addition of the cellulase. A pH in the range from 4 to 6 and a temperature in the range from 20 to 70°C may often be found convenient, in accordance with the recommendations of the enzyme supplier. The pH and temperature of the liquor during the defibrillation step (C) should be in accordance with the recommendations of the cellulase supplier. It was surprising to find that the defibrillation step (C) in the method of the invention proceeds normally, because it is often observed that the presence of extraneous substances can

degrade enzyme activity.

[0029] The liquor may be made alkaline during step (D) in order to improve dye fixation.

[0030] A rinsing step and a softening step (involving the application of a fabric softener) preferably follow the denaturing and re fibrillation step (D) and precede the drying step (E) as described hereinabove.

[0031] Removal of unfixed reactive dyestuff after a dyeing operation commonly requires that the dyed fabric be washed several times with water. It was surprising to find that in this embodiment of the method of the invention the cellulase treatment step serves to perform this function, even though it utilises exhausted dye liquor containing a cellulase rather than clean water.

[0032] The method of the invention occupies typically 8-9 hours and requires the use of two or three liquor baths. It thus offers considerable savings in both time and water usage compared with conventional techniques.

[0033] Comparative experiments on fabrics of cotton or viscose surprisingly gave less satisfactory results. No fewer than five additional rinsing steps were required (on some kinds of conventional processing equipment) after the enzyme treatment step when using such fabrics in order to remove unfixed dye from the system. Furthermore, depth of shade was lower and fastness was inferior to that obtained on lyocell. It will also be appreciated that cotton and viscose fibres exhibit little or no tendency to fibrillation during textile processing.

[0034] The method of the invention may be used to dye blends of lyocell and polyester or of polynosic and polyester, in which case the dyebath will contain both reactive and disperse dyestuffs. The method of the invention may also be used to dye blends of lyocell and polyamide or of polynosic and polyamide, in which case the dyebath will contain both reactive and acid dyestuffs; in the case of fixation under alkaline conditions, care should be taken to select acid dyestuffs with adequate fastness to alkali.

[0035] The method of the invention has the advantage that the number of cold-treatment steps is minimised. It has been found that rinsing or other treatment of lyocell fabrics with cold water during dyeing and finishing may lead to fabric damage (i.e. excessive fibrillation, including the formation of crease marks and white-line damage).

[0036] Fibrillation of lyocell fabrics may be measured by the following Test Method:-

Test Method

[0037] There is no universally accepted standard for assessment of fibrillation, and the following method was used to assess Fibrillation Index (F.I.). Samples of fibre were arranged into a series showing increasing degrees of fibrillation. A standard length of fibre from each sample was then measured and the number of fibrils (fine hairy spurs extending from the main body of the fibre) along the standard length was counted. The length of each fibril was measured, and an arbitrary number, being the number of fibrils multiplied by the average length of each fibril, was determined for each fibre. The fibre exhibiting the highest value of this product was identified as being the most fibrillated fibre and was assigned an arbitrary Fibrillation Index of 10. A wholly unfibrillated fibre was assigned a Fibrillation Index of zero, and the remaining fibres were evenly ranged from 0 to 10 based on the microscopically measured arbitrary numbers.

[0038] The measured fibres were then used to form a standard graded scale. To determine the Fibrillation Index for any other sample of fibre, five or ten fibres were visually compared under the microscope with the standard graded fibres. The visually determined numbers for each fibre were then averaged to give a Fibrillation Index for the sample under test. It will be appreciated that visual determination and averaging is many times quicker than measurement, and it has been found that skilled fibre technologists are consistent in their rating of fibres.

[0039] Fibrillation Index of fabrics can be assessed on fibres drawn from the surface of the fabric. Woven and knitted fabrics having F.I. of more than about 2.0 to 2.5 are normally found to exhibit an unsightly appearance.

[0040] The invention is illustrated by the following Examples, in which parts and proportions are by weight unless otherwise specified:-

Example 1

[0041] A woven lyocell fabric (plain weave, basis weight 190 grams per square metre, of 1/20s cotton count (30 tex) ring-spun 100% lyocell yarn) (Tencel, Trade Mark of Acordis Fibres (Holdings) Limited - formerly known as Courtaulds Fibres (Holdings) Limited) was scoured in open width to remove residual size. The fabric was circulated through dye liquor at 50°C in a Thies Rototumbler (Trade Mark) jet-processing machine (modified to permit dyeing). The dye liquor contained 3% Kayacelon React Red CN-3B (Trade Mark) and 80 grams per litre Glauber's Salt. The temperature of the liquor was raised at 2°C/min to a final temperature of 130°C, and dyeing was continued for 60 min at this temperature. The dyed fabric exhibited primary fibrillation and had a measured F.I. of 6.6. The exhausted dye liquor was cooled to 55°C while the fabric continued to circulate through the machine. The pH of the liquor was adjusted to 4.5-5 by addition of 3 ml/l Sandacid BS (Trade Mark) buffer solution. 3% Primafast 100 (Trade Mark of Genencor) liquid cellulase preparation was added to the liquor and the fabric circulated for 1 hr, after which its F.I. was measured to be 1.0. Liquor temperature was raised to 95°C and circulation continued for 45 min in order to denature the cellulase and

to induce secondary fibrillation. The liquor was cooled to 45°C and 3% Sandoperm MEJ (Trade Mark of Clariant AG) (fabric softener) added, after which circulation was continued for 20 minutes. The liquor was then discarded, and the fabric was dried by continued circulation through the machine using hot air. Circulation was continued for 1 hr after the fabric was dry. Such dry tumbling lifts the pile of the fabric to give more desirable visual appearance and handle and serves to remove loose fibres remaining from the defibrillation step.

[0042] In a control experiment, the woven lyocell fabric was scoured in preparation for dyeing as previously described. The fabric was dyed with a 3% solution of the reactive dyestuff Procion Red H-EXL (Trade Mark of BASF AG) containing salt and alkali in accordance with the recommendations of the dyestuff manufacturer, in the modified Thies Rototumbler previously mentioned. After dyeing, residual dyestuff was removed from the fabric by seven washings in accordance with the recommendations of the dyestuff manufacturer: cold, hot, warm, boil, hot, warm, cold. The fabric was then treated with a 3% solution of the cellulase Primafast 100 for 1 hr at 55°C. The temperature of the solution was raised to 85°C and circulation continued to denature the enzyme and to induce secondary fibrillation. The fabric was rinsed and a softener solution applied to it. The fabric was then dried and tumbled as described above.

[0043] Fabric treated according to the method of the invention and the control fabric both exhibited excellent wash fastness in cross-staining experiments against a variety of fabrics. Stability to laundering was good and two fabrics gave comparable results. Both fabrics exhibited a peach-touch finish. The crease recovery of the two fabrics was similar. Measured tensile properties and abrasion results are given in Table 1.

Table 1

		Control	Invention
Tensile strength N	- warp	652	831
	- weft	271	439
Extensibility %	- warp	13	14
	- weft	8	10
Seam strength daN	- warp	124	174
	- weft	195	229
Martindale abrasion rev	- dry	11500	12000
	- wet	775	700
Stoll abrasion rev	- warp	194	596
	- weft	218	614

[0044] The tensile strength, seam strength and Stoll abrasion resistance of the sample treated according to the invention were markedly better than those of the control sample.

Example 2

[0045] Lyocell fabric (as used in Example 1, except that the basis weight was 180 g/m²) was scoured and bleached in open width. Subsequent wet processing was performed in stainless steel pots (300 ml capacity) housed in a Zeltex Vistacolor (Trade Mark) dyeing machine, with a liquor-to-goods ratio of 20:1 (fabric sample weight 4 g). The fabric was dyed using a dye liquor containing 2% owf (on weight of fabric) of a Procion H-EXL (Trade Mark) dyestuff (Procion Blue H-EXL, Procion Crimson H-EXL, or Procion Yellow H-EXL), 40 g/l sodium chloride and a phosphate buffer to control liquor pH at 7.4 but no added alkali at this stage. The temperature of the dye liquor was maintained at 50°C for 10 min, then raised to 130°C at a rate of 2°C/min and maintained at the latter temperature for 30 min. The dye liquor was then cooled to 80°C, 15 g/l sodium carbonate was added, and the temperature of the liquor was maintained at the latter figure for 30 min. The dye liquor was discarded, and the fabric was rinsed in water at 80°C for 15-30 min. The fabric was then treated with a solution containing 2% owf of a cellulase, the pH of the solution being adjusted to 4.5 by addition of acetic acid. The following cellulases were tested: Apolase AC conc (Trade Mark of Brookstone), Primafast 100 (Trade Mark of Genencor) and Cellusoft Plus L (Trade Mark of Novo Nordisk). The cellulase treatment was performed at 50°C for 45 min. The fabric was then washed and dried.

[0046] Colorimetric studies showed that the enzyme treatment removed unfixed dyestuff from the fabric. All samples exhibited good depth of shade and excellent wash fastness. All samples exhibited a greater depth of shade than comparative samples dyed in accordance with the recommendations of the dyestuff manufacturer. The samples exhibited a moderately developed peach-touch finish; this was a consequence of the small-scale equipment employed, and it is believed that processing on conventional mill-scale equipment would have permitted development of a full peach-touch finish.

Example 3

[0047] Lyocell fabric (as used in Example 2) was scoured and bleached in open width. Subsequent wet processing was performed in stainless steel pots (300 ml capacity) housed in a Zeltex Vistacolor (Trade Mark) dyeing machine, with a liquor-to-goods ratio of 20:1 (fabric sample weight 4 g). The fabric was dyed using a dye liquor containing 2% owf of a Procion H-EXL (Trade Mark) dyestuff (Procion Blue H-EXL, Procion Crimson H-EXL, or Procion Yellow H-EXL), 40 g/l sodium chloride and a phosphate buffer to control liquor pH at 7.4. The temperature of the dye liquor was maintained at 50°C for 10 min, then raised to 130°C at a rate of 2°C/min and maintained at the latter temperature for 30 min. The dye liquor was then cooled and acidified, and 2% owf cellulase was added to the liquor. In one experiment, the liquor was cooled to 50°C, its pH was adjusted to 4.5, and the cellulase Cellusoft Plus L (Trade Mark of Novo Nordisk) was added. In another experiment, the liquor was cooled to 60°C, its pH was adjusted to 6, and the cellulase Forylase SW150 (Trade Mark of Henkel) was added. The fabric was maintained in contact with the liquor for 30 min, whereupon inspection showed that primary fibrils had been removed. The temperature was raised to 80°C, and then sufficient sodium carbonate was added to raise the pH to 11-12. After a further 30 min, the fabric was washed with 2 g/l Lanapex R (Trade Mark of ICI Surfactants) for 30 min at the boil, rinsed in cold water and dried.

[0048] The treated fabric exhibited excellent wash fastness and a similar finish to those of Example 2. In comparison with fabric treated with cellulases under near-neutral conditions, these fabrics treated with cellulases under acidic conditions exhibited a slightly lower depth of shade.

Example 4

[0049] A woven lyocell fabric (2x1 twill, basis weight 230 grams per square metre; Tencel) was prepared in open width in conventional manner. The fabric was circulated in rope form through dye liquor at 60°C in a Then AFS (Trade Mark) jet dyeing machine. The dye liquor contained a conventional dyebath lubricant, 40 g/l Glauber's salt and a mixture of reactive dyestuffs (1.8% Procion Yellow H-EXL, 0.22% Procion Crimson H-EXL, 1.8% Procion Navy H-EXL) (Procion is a Trade Mark of BASF). The temperature of the liquor was raised to 130°C at 2°C/min and maintained there for 10 minutes. The liquor was cooled to 100°C at 1°C/min and then to 55°C at 1.5°C/min. 1 g/l of buffer solution was added to adjust pH to 4.5-5.5, and next 2 g/l Primafast 100 (Trade Mark of Genencor) cellulase enzyme was added. The fabric was circulated for 90 minutes before raising the temperature to 78°C at 1°C/min, after which 20 g/l soda ash was added as dye fixative. The fabric was circulated for a further 60 minutes. The liquor was discarded, and the fabric was rinsed in 40°C water. The fabric was washed in 95°C water for 10 minutes and cooled to 60°C, after which the wash water was discarded. The fabric was rinsed at 70°C for 10 minutes in water containing 1 g/l Duralkan CTI (Trade Mark of Thor) and finally rinsed in cold water. The fabric was removed from the dyeing machine, dewatered and padded with conventional fabric softeners. It was loaded onto a Biancalani Airo (Trade Mark) tumbler for drying. Beating was continued in hot air in the tumbler for 15 minutes after drying was complete. The resulting fabric exhibited a peach-touch handle and appearance. Process time on the dyeing machine was 8.5 hours, compared with over 12 hours in the conventional process. The amount of water used was also considerably less.

Examples 5-8

[0050] Similar procedures to Examples 1-4 may be carried out on polynosic fabrics to achieve similar results.

Claims

1. A method for dyeing and finishing a lyocell fabric or a polynosic fabric including the steps of:

(1) applying to the fabric in rope form a liquor containing in solution a reactive dyestuff for cellulose, under conditions such that both (i) a high degree of exhaustion of the dyestuff onto the fabric is achieved and (ii) fibrillation of the fabric occurs, thereby providing a dyed fabric exhibiting primary fibrillation;

(2) applying to the fabric in rope form with no more than a single intermediate rinse a solution of a cellulase, and maintaining the solution in contact with the fabric under conditions effective to remove primary fibrils therefrom by the action of the cellulase, thereby providing a dyed fabric exhibiting a low degree of fibrillation;

(3) raising the temperature of the solution whilst it is maintained in contact with the fabric, whereby both (i) the cellulase is denatured and (ii) secondary fibrillation is induced in the fabric, thereby providing a dyed fabric exhibiting secondary fibrillation; and

(4) drying the fabric.

2. A method according to claim 1, further **characterised in that** there is no intermediate rinse between steps (1) and (2).

3. A method according to claim 1 or claim 2, further **characterised in that** step (4) is performed in a tumbling machine, and **in that** tumbling is continued therein after drying of the fabric is complete in order to lift the pile of the fabric and to remove loose fibres therefrom.

4. A method according to any one of the preceding claims, further **characterised in that** step (1) involves the following steps in sequence:

(i) applying to the fabric a liquor containing in solution a reactive dyestuff for cellulose, the solution containing no added alkali;

(ii) maintaining the liquor in contact with the fabric at a temperature in the range from 100 to 150°C until fibrillation has occurred;

(iii) cooling the liquor to a temperature within the range from 40 to 100°C;

(iv) adding alkali to the liquor; and

(v) maintaining the liquor in contact with the fabric in order to complete fixation of the dyestuff onto the fabric.

5. A method according to claim 4, further **characterised in that** the pH of the dye liquor in step (i) is controlled by addition of a pH buffer.

6. A method for dyeing and finishing a lyocell fabric or a polynosic fabric including the steps of:

(A) applying to the fabric in rope form a liquor containing a reactive dyestuff in solution, the dyestuff being capable of reacting with cellulose in the absence of added alkali, under conditions such that both (i) a high degree of exhaustion of the dyestuff onto the fabric is achieved and (ii) fibrillation of the fabric occurs, thereby providing a dyed fabric exhibiting primary fibrillation;

(B) adding a cellulase to the liquor;

(C) maintaining the liquor in contact with the fabric under conditions effective to remove primary fibrils therefrom by the action of the cellulase, thereby providing a dyed fabric exhibiting a low degree of fibrillation;

(D) raising the temperature of the liquor whilst it is maintained in contact with the fabric, whereby both (i) the cellulase is denatured and (ii) secondary fibrillation is induced in the fabric, thereby providing a dyed fabric exhibiting secondary fibrillation; and

(E) drying the fabric.

7. A method according to claim 6, further **characterised in that** step (A) is performed at a temperature in the range from 100 to 150°C.

8. A method according to any one of the preceding claims, further **characterised in that** it includes a rinsing step between steps (3) and (4) or between steps (D) and (E) as the case may be.

9. A method according to any one of the preceding claims, further **characterised in that** a fabric softener is applied to the fabric immediately prior to step (4) or step (E) as the case may be.

Patentansprüche

1. Verfahren zur Veredlung einer Lyocell-Ware oder einer Polynosic-Ware, bei dem man

(1) auf die in Strangform vorliegende Ware eine einen Reaktionsfarbstoff für Cellulose in Lösung enthaltende Flotte so aufträgt, daß sowohl (i) der Farbstoff hochgradig auf die Ware aufzieht als auch (ii) eine Fibrillierung der Ware erfolgt, wodurch man eine gefärbte Ware mit Primärfibrillation erhält,

(2) auf die in Strangform vorliegende Ware nach höchstens einer einzigen Zwischenspülung eine Lösung einer Cellulase aufträgt und die Lösung so in Kontakt mit der Ware verweilt, daß die primären Fibrillen durch die Wirkung der Cellulase von der Ware entfernt werden, wodurch man eine gefärbte Ware mit einem geringen Fibrillierungsgrad erhält,

(3) die Temperatur der Lösung in Kontakt mit der Ware erhöht und dadurch sowohl (i) die Cellulase denaturiert als auch (ii) in der Ware eine sekundäre Fibrillierung ausgelöst wird, wodurch man eine gefärbte Ware mit Sekundärfibrillation erhält, und

(4) die Ware trocknet.

2. Verfahren nach Anspruch 1, ferner **dadurch gekennzeichnet, daß** zwischen den Schritten (1) und (2) keine Zwischenspülung erfolgt.

3. Verfahren nach Anspruch 1 oder Anspruch 2, ferner **dadurch gekennzeichnet, daß** man Schritt (4) in einem Tumbler durchführt und auch nach dem vollständigen Trocknen der Ware weiter taumelt, um den Flor auf der Ware aufzurichten und alle darin schlecht eingebundenen Fasern davon zu entfernen.

4. Verfahren nach einem der vorhergehenden Ansprüche, ferner **dadurch gekennzeichnet, daß** man in Schritt (1) nacheinander

(i) auf die Ware eine einen Reaktionsfarbstoff für Cellulose in Lösung enthaltende Flotte aufträgt, wobei die Lösung keinen Zusatz von Alkali enthält,

(ii) die Flotte bei einer Temperatur im Bereich von 100 bis 150°C solange in Kontakt mit der Ware verweilt, bis eine Fibrillierung erfolgt ist,

(iii) die Flotte auf eine Temperatur im Bereich von 40 bis 100°C abkühlt,

(iv) die Flotte mit Alkali versetzt und

(v) die Flotte in Kontakt mit der Ware verweilt, um die Farbstofffixierung auf der Ware zu vervollständigen.

5. Verfahren nach Anspruch 4, ferner **dadurch gekennzeichnet, daß** man in Schritt (i) den pH-Wert der Färbeflotte durch Zusatz eines pH-Puffers einstellt.

6. Verfahren zur Veredelung einer Lyocell-Ware oder einer Polynosic-Ware, bei dem man

(A) auf die in Strangform vorliegende Ware eine einen auch alkalifrei mit Cellulose reaktionsfähigen Reaktionsfarbstoff in Lösung enthaltende Flotte so aufträgt, daß sowohl (i) der Farbstoff hochgradig auf die Ware aufzieht als auch (ii) eine Fibrillierung der Ware erfolgt, wodurch man eine gefärbte Ware mit Primärfibrillation erhält,

(B) die Flotte mit einer Cellulase versetzt,

(C) die Flotte in Kontakt mit der Ware so verweilt, daß die primären Fibrillen durch die Wirkung der Cellulase von der Ware entfernt werden, wodurch man eine gefärbte Ware mit einem geringen Fibrillierungsgrad erhält,

(D) die Temperatur der Flotte in Kontakt mit der Ware erhöht und dadurch sowohl (i) die Cellulase denaturiert als auch (ii) in der Ware eine sekundäre Fibrillierung ausgelöst wird, wodurch man eine gefärbte Ware mit Sekundärfibrillation erhält, und

(E) die Ware trocknet.

7. Verfahren nach Anspruch 6, ferner **dadurch gekennzeichnet, daß** Schritt (A) bei einer Temperatur im Bereich von 100 bis 150°C erfolgt.
8. Verfahren nach einem der vorhergehenden Ansprüche, ferner **dadurch gekennzeichnet, daß** zwischen den Schritten (3) und (4) bzw. (D) und (E) eine Zwischenspülung erfolgt.
9. Verfahren nach einem der vorhergehenden Ansprüche, ferner **dadurch gekennzeichnet, daß** man unmittelbar vor Schritt (4) bzw. (E) auf die Ware ein Weichgriffmittel aufträgt.

Revendications

1. Procédé de teinture et de finition d'une étoffe lyocell ou d'une étoffe polynosique comprenant les étapes suivantes :

(1) application à l'étoffe sous forme de boyaux d'une liqueur contenant en solution un colorant réactif pour la cellulose, dans des conditions telles qu'à la fois (i) on obtienne un degré élevé de fixation du colorant sur l'étoffe et (ii) qu'il se produise une fibrillation de l'étoffe, ce qui permet d'obtenir une étoffe teinte présentant une fibrillation primaire;

(2) application à l'étoffe sous forme de boyaux, avec pas plus d'un seul rinçage intermédiaire, d'une solution d'une cellulase et maintien de la solution en contact avec l'étoffe dans des conditions efficaces pour en éliminer les fibrilles primaires par l'action de la cellulase, ce qui permet d'obtenir une étoffe teinte présentant un faible degré de fibrillation;

(3) élévation de la température de la solution tout en la maintenant en contact avec l'étoffe de manière qu'à la fois (i) la cellulase soit dénaturée et (ii) qu'une fibrillation secondaire soit induite dans l'étoffe, ce qui permet d'obtenir une étoffe teinte présentant une fibrillation secondaire; et

(4) séchage de l'étoffe.

2. Procédé selon la revendication 1, **caractérisé en outre en ce qu'il n'y a pas de rinçage intermédiaire** entre les étapes (1) et (2).

3. Procédé selon la revendication 1 ou 2, **caractérisé en outre en ce que** l'étape (4) est effectuée dans une machine à culbutage et **en ce que** le culbutage y est continué après que le séchage de l'étoffe a été terminé de manière à soulever le poil de l'étoffe et à éliminer les fibres détachées.

4. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en outre en ce que** l'étape (1) implique les étapes suivantes en séquence :

(i) application à l'étoffe d'une liqueur contenant en solution un colorant réactif pour la cellulose, la solution ne contenant d'alcali ajouté;

(ii) maintien de la liqueur en contact avec l'étoffe à une température dans la plage de 100°C à 150°C jusqu'à ce qu'il se soit produit une fibrillation;

(iii) refroidissement de la liqueur à une température dans la plage de 40°C à 100°C;

(iv) addition d'alcali à la liqueur; et

(v) maintien de la liqueur en contact avec l'étoffe pour terminer la fixation du colorant sur l'étoffe.

5. Procédé selon la revendication 4, **caractérisé en outre en ce que** le pH de la liqueur de colorant à l'étape (i) est réglé par addition d'un tampon de pH.

6. Procédé de teinture et de finition d'une étoffe lyocell ou d'une étoffe polynosique, comprenant les étapes suivantes :

(A) application à l'étoffe sous forme de boyaux d'une liqueur contenant un colorant réactif en solution, le colorant étant capable de réagir avec la cellulose en l'absence d'alcali ajouté, dans des conditions telles qu'à la fois (i) on obtienne un degré élevé de fixation du colorant sur l'étoffe et (ii) qu'il se produise une fibrillation de l'étoffe, ce qui permet d'obtenir une étoffe teinte présentant une fibrillation primaire;

(B) addition d'une cellulase à la liqueur;

(C) maintien de la liqueur en contact avec l'étoffe dans des conditions efficaces pour en éliminer les fibrilles primaires par l'action de la cellulase, ce qui permet d'obtenir une étoffe teinte présentant un faible degré de fibrillation;

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(D) élévation de la température de la liqueur tout en la maintenant en contact avec l'étoffe, de sorte qu'à la fois (i) la cellulase soit dénaturée et (ii) qu'une fibrillation secondaire soit induite dans l'étoffe, ce qui permet d'obtenir une étoffe teinte présentant une fibrillation secondaire; et
(E) séchage de l'étoffe.

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7. Procédé selon la revendication 6, **caractérisé en outre en ce que** l'étape (A) est effectuée à une température dans la plage de 100°C à 150°C.

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8. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en outre en ce qu'il** comprend une étape de rinçage entre les étapes (3) et (4) ou entre les étapes (D) et (E), selon le cas.

9. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en outre en ce qu'un** adoucisseur de tissu est appliqué à l'étoffe immédiatement avant l'étape (4) ou l'étape (E), selon le cas.

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