(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
16 September 2004 (16.09.2004)

PCT

(10) International Publication Number
WO 2004/079082 A1

(51) International Patent Classification[7]: D06M 13/188, 23/00, D06P 5/20, 3/60

(21) International Application Number: PCT/GB2004/000833

(22) International Filing Date: 1 March 2004 (01.03.2004)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
0304919.4 4 March 2003 (04.03.2003) GB

(71) Applicant *(for all designated States except US)*: **TENCEL LIMITED** [GB/GB]; 1 Holme Lane, Spondon, Derby DE21 7BP (GB).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: **TAYLOR, James, Martin** [GB/GB]; 42 Linacres Drive, Chellaston, Derby DE73 1XH (GB). **BRIGGS, Nigel, Philip** [GB/GB]; 41 Belfiel Road, Etwall, Derby DE65 6JN (GB).

(74) Agent: **HALE, Stephen, Geoffrey**; Bromhead Johnson, Kingsbourne House, 229-231 High Holborn, London WC1V 7DP (GB).

(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Declaration under Rule 4.17:

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii)) for the following designations AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG)*

Published:

— *with international search report*
— *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: PROCESS FOR PRODUCING A DYED AND FINISHED LYOCELL FABRIC

(57) Abstract: A dyed and finished lyocell fabric having a clean, soft-touch finish is produced by a process in which fibrillation is induced in a wet processing step prior to dyeing. This step is carried out under pressure at high temperature using an aqueous carboxylic acid solution and enables the io unwanted longer fibre ends produced by fibrillation to be removed. Acetic acid and formic acid are the preferred carboxylic acids. Processing may be carried out on a fabric rope using an air-jet dyeing machine both for the fibrillation and the dyeing steps.

WO 2004/079082 A1

PROCESS FOR PRODUCING A DYED AND FINISHED LYOCELL FABRIC

Technical Field

This invention relates to the dyeing and finishing of a lyocell fabric.

In this specification, the term "lyocell fabric" means a fabric woven or knitted from staple fibre yarns comprising lyocell fibres. Such yarns may comprise lyocell fibres alone or they may comprise a blend of lyocell fibres with fibres of one or more other fibre types, either cellulosic or non-celluosic, such as cotton, viscose, linen, polyester and nylon. Furthermore, the fabric may additionally include yarns which do not incorporate lyocell fibres, for example yarns of the other fibre types referred to and blends thereof.

Lyocell fibres are produced by extrusion of a solution of cellulose through a spinning jet into a coagulation bath by a process known as solvent spinning. Thus, an alternative name for lyocell fibres is solvent-spun cellulose fibres. Such a process is described in US-A-4,246,221 and uses as the solvent an aqueous tertiary amine N-oxide, particularly N-methylmorpholine N-oxide. Lyocell fibres are distinguished from other man-made cellulose fibres which are produced by forming the cellulose into a soluble chemical derivative and then extruding a solution of this derivative into a bath which regenerates the extrudate as cellulose fibres; viscose fibres, including the high strength modal types, are produced in this latter way.

Background Art

Lyocell fibres are known to have a tendency to fibrillate during vigorous dyeing and finishing processes, and there

have been a number of methods of dealing with this phenomenon. Where fibrillation is desired to be avoided, then dyeing and finishing of lyocell fabrics is carried out using relatively gentle processing such as pad-dyeing of open-width fabric, and a resin-finishing treatment using a cross-linking agent is carried out on the dyed fabric to protect the fibres against fibrillation in subsequent laundering processes.

Another method of dealing with this fibrillation tendency of lyocell fibres is to treat the fibres so as to remove the relatively long protruding fibre ends which are formed in the first stage of the fibrillation process (so-called "primary fibrillation") and which otherwise produce a hairy effect, often matted, on the surface of the fabric and thereby disfigure its appearance. On the other hand, the development of the shorter fibrils which are formed in the fibrillation process (so-called "secondary fibrillation") is encouraged. These shorter fibrils create a surface finish which is characterised as being "clean", in the sense of being substantially free from producing a hairy effect, and as having a soft touch imparted by the shorter fibrils on the surface and referred to as a "soft-touch finish". When the shorter fibrils are sufficiently developed, then the soft touch of the fabric surface is more pronounced and the soft-touch finish is referred to as a "peach-touch finish".

An example of this approach for dealing with fibrillation tendency is described in WO-A-95/30043 and involves removing the product of primary fibrillation formed during a dyeing process by a post-treatment with an acid catalyst used in conjunction with an optional cross-linking agent such as an N-methylol resin. Another example is described in WO-A-97/30204, where the lyocell fabric before dyeing is given a pre-treatment with an aqueous solution of an oxidising agent such as sodium hypochlorite or hydrogen peroxide at elevated temperatures. A further example is

described in GB-A-2,314,568 and involves use of the dyeing machine itself, for example a jet dyeing machine, to subject the lyocell fabric to an extended treatment with a solution of a strong mineral acid such as sulphuric acid for 30 to 120 minutes before rinsing the fabric and then commencing the dyeing sequence in the machine. Each of these processes has been difficult to control to produce consistent results and none of them has been taken through into full-scale commercial operation.

A different approach to the problem is described in WO-A-02/103104 and involves a pre-treatment of the lyocell fabric before the fibrillation-inducing step of dyeing. This pre-treatment comprises impregnation of the fabric with an aqueous solution of an acid or acid donor such as citric acid or magnesium chloride, followed by heat treatment of the impregnated fabric in a gaseous atmosphere at 120°C to 220°C to activate the action of the acid or acid donor. This process is effective, but the requirement to use the gaseous heat treatment, which usually means using a stenter oven, is a step which many fabric processors would prefer to avoid.

One process which has been used successfully in commercial processing involves a post-treatment of a lyocell fabric which has been subjected to a wet processing operation, to cause fibrillation. The protruding fibre ends produced in primary fibrillation are removed in this post-treatment by applying to the fabric a solution of a cellulase enzyme. The desired secondary fibrillation is developed in subsequent processing, for example in the dyeing process itself (if later) or in subsequent washing and drying steps using rotary tumbling machines, to produce a soft-touch finish on the surface of the fabric.

Cellulase enzyme treatments are successful in removing the long fibre ends produced in primary fibrillation from the surface of the fabric, but they are expensive both in

terms of material costs and in terms of processing time.

Disclosure of the Invention

The present invention provides a process for producing a dyed and finished lyocell fabric having a clean, soft-touch finish, comprising subjecting a lyocell fabric to a wet processing treatment using mechanical action to induce fibrillation on the surface of the fabric and then dyeing the fabric before giving it a tumbling treatment to develop the clean, soft-touch finish, characterised in that the fibrillation-inducing step is carried out at above atmospheric pressure, using as the wet processing liquid an aqueous solution of a carboxylic acid which is at a temperature which is above the boiling point of the solution at atmospheric pressure.

The lyocell fabric preferably is processed in fabric rope form. It is also possible to process the fabric in the form of piece goods or garments. In each case, the equipment used should be capable of being pressurised to allow the required temperature to be reached in the fibrillation-inducing step. Preferably, the same equipment is used in both the fibrillation-inducing step and the dyeing step.

An example of equipment which may be used in the fibrillation-inducing step comprises a jet dyeing machine in which a rope of fabric is transported cyclically through a processing liquid under the action of air or liquid jets which effect mechanical action on the rope of fabric. This action is in the form of bending and twisting forces and abrasive and impact forces as the rope is propelled forcibly against machine surfaces. The same jet dyeing machine can be used for the subsequent dyeing step.

It is preferred to use air-jet dyeing machines. Suitable commercial machines include the Thies Airstream, the Thies Luft Roto, the Hisaka AJ-1, the Krantz Aerodye and the Then

AFS.

When the lyocell fabric is in the form of piece goods or garments, then the equipment used may be a conventional closed dyeing vessel used for that purpose, such as a rotary-drum dyeing machine. Rotation of the machine parts causes mechanical forces to be applied to the wet garments or piece goods within the machine to cause the desired fibrillation.

The wet processing liquid used in the fibrillation-inducing step is an aqueous solution of a carboxylic acid. This is preferably a lower aliphatic carboxylic acid, for example of up to 6 or 8 carbon atoms, suitable compounds including acetic acid, formic acid, citric acid and tartaric acid. Acetic acid and formic acid are preferred compounds.

The concentration of the carboxylic acid in the aqueous solution used as the wet processing liquid, the temperature and the time of treatment are all interdependent for best results in the fibrillation-inducing step, lower concentrations being usable as treatment temperature is increased and treatment time is extended. In general, it is desirable to keep treatment temperatures and treatment times within reasonable limits, for commercial and operational reasons. A particularly suitable temperature for the treatment in the fibrillation-inducing step is in the range 120°C to 140°C, with a suitable treatment time at those temperatures being in the range 30 to 80 minutes. Under these conditions of treatment, the concentration of the carboxylic acid in the wet processing liquid is generally in the range 0.2 to 50 g/l (grams per litre).

Within this broader range, the identity of the particular carboxylic acid influences the concentrations used. For acetic acid, a particularly suitable range of concentration is 2 to 50 g/l, preferably 3 to 10 g/l. For formic acid, a particularly suitable range of concentration is 0.2 to 4 g/l, preferably 0.4 to 1.2 g/l.

Acetic acid and formic acid are both very effective in the process of the invention. In terms of effluent treatment, however, there is an advantage in using formic acid because of its lower chemical oxygen demand.

The overall treatment time will include a start-up period during which the wet processing liquid is heated to bring it up to the desired treatment temperature. The pressure in the vessel will increase accordingly to allow that temperature to be achieved. There is also a shutting-down period at the end of the treatment when the wet processing liquid is cooled down and the fabric is rinsed with water prior to the dyeing step. These periods together usually will be at least as long as the period of treatment itself, so that the overall time taken for this step may be 60 to 160 minutes.

The mechanical action exerted on the wet fabric in this step causes fibrillation to occur on the fabric surface. This fibrillation is of the so-called primary fibrillation type in which relatively long protruding fibre ends are produced to give what would be an unsightly, hairy effect on the fabric surface if these long fibre ends were to remain in place. The effect of the simultaneous high temperature treatment with the carboxylic acid is to substantially weaken the strength of these fibre ends, so that they are removed from the fabric surface during processing.

It appears that many of the fibre ends are removed during the carboxylic acid treatment itself, with any remaining fibre ends being rubbed off the fabric surface during the subsequent dyeing and finishing steps. Usually, any fibre ends remaining after dyeing are in the form of unattached lint, which is removed from the fabric surface during the fabric tumbling step.

If the yarns of the lyocell fabric have been sized or

lubricated to facilitate weaving or knitting, then the fabric may be subjected to a desizing or scouring operation carried out prior to the process of the invention. This desizing or scouring operation may be a conventional operation in which the fabric is treated with a scouring liquor to remove the size or lubricant.

Another possible treatment of the lyocell fabric is a so-called causticising treatment with an aqueous sodium hydroxide solution. If appropriate, this should be carried out before the process of the invention but after any de-sizing or scouring operation. Causticising is carried out to improve the flexibility of the fabric in the wet state. A process for its application to lyocell fabrics is described in EP-A-0,749,505. Causticising also enhances dyeability of the fabric, which may be depressed by the carboxylic acid treatment, and has the further effect that the fabric is tightened in structure, which helps to reduce any propensity for shrinkage in later wet processing. After causticising, the fabric should be thoroughly rinsed with hot and then cold water to remove residual caustic soda.

After the fabric has been washed at the end of the fibrillation-inducing step, it is dyed, preferably using the same equipment. In the case of processing the fabric in fabric rope form, this equipment is preferably a jet dyeing machine, more preferably an air-jet dyeing machine. All that is then required is for the dyebath to be introduced into the machine and the dye cycle to be started and run. This dye cycle may be a conventional dyeing process for lyocell fabric using the usual operating conditions and the usual dye and dye liquor recipes, including those based on direct dyes, vat dyes, sulphur dyes and reactive dyes.

With lyocell fabrics comprising a blend of lyocell fibres and polyester fibres, the polyester fibres are dyed using a disperse dye and the standard procedure is to carry this out as a separate operation from the dyeing of the lyocell

fibres. In the process of the invention, it has been found unnecessary to have this disperse dyeing operation as an additional step in the process because it can be combined with the fibrillation-inducing step. The resulting presence in the dyebath of higher levels of carboxylic acid than are normally encountered in the disperse dyeing of polyester fibres does not prevent good quality, even dyeing of the polyester component of the fabric. Conversely, the presence of the disperse dye liquor does not interfere with the functioning of the fibrillation-inducing step.

After being washed to remove any unfixed dye, the dyed fabric may be given conventional finishing treatments including application of a soft finish. This may be carried out after the dyeing and washing processes without the need for any intermediate drying of the fabric.

At this stage, the surface of the dyed fabric does not yet have the desired clean, soft-touch finish. It remains flat and uneven in appearance, possibly with some adherent lint. In order to develop the desired finish, the fabric is given a tumbling treatment, sometimes referred to as a beating treatment, which raises the relatively short fibres of so-called secondary fibrillation in an even nap over the fabric surface. This treatment preferably is carried out as a dry treatment, i.e. without added liquid, and it may be the step in which the fabric is given its final drying.

Garments and piece goods may be given a tumbling treatment in a rotary tumbling machine. Fabric in fabric rope form may be given a tumbling treatment in a fabric rope tumbler such as a Biancalani Airo tumbling machine.

Tumbling treatment times for a fabric rope are usually no longer than 30 to 50 minutes using air temperatures of 50°C to 150°C.

The dry lyocell fabric produced by the process of the invention is a dyed fabric having a clean, soft-touch finish. The uniform, soft nap on the fabric surface imparts an attractive handle to the fabric and a subtle appearance, sometimes referred to as a frosted effect. When the nap is developed to a higher level, then the fabric surface may be characterized as having a peach-touch finish.

As mentioned earlier, the lyocell fabric may be a woven fabric or a knitted fabric. In both cases, the process of the invention preferably is carried out on the fabric in fabric rope form rather than in open width. In contrast, any pre-treatments required on woven fabrics, such as desizing, scouring and causticising, are best carried out in open width. Where scouring of knitted fabrics is required, however, it may be carried out with the fabric in fabric rope form. This makes it possible to treat a knitted fabric in the same fabric rope form through the whole process sequence, which is a considerable logistical advantage.

The invention is illustrated by the following Examples. In all of the Examples, the lyocell fibres of the processed lyocell fabrics were TENCEL (Registered Trade Mark) fibres produced by Tencel Limited.

Example 1

A lyocell fabric comprised a woven fabric of basis weight 179 gsm (grams per square metre) constructed in a 3 by 1 twill weave from yarn of count 1/30s Ne. The yarn comprised 100 per cent TENCEL lyocell fibres.

The fabric was scoured in open width using an aqueous scour bath containing a non-ionic detergent and sodium carbonate and at a temperature of 90°C.

The desized fabric was then formed into a fabric rope, which was loaded into an air-jet dyeing machine. The machine was actually a Thies Rototumbler machine, which had been adapted by Thies to allow dyeing to be carried out in the machine in addition to non-dyeing tumbling processes, so that the machine is more versatile for trial work. The machine was rated for use at pressures above atmospheric pressure.

The treatment liquid for use in the fibrillation-inducing step was then introduced into the machine. It was an aqueous solution comprising 6.4 g/l acetic acid (100%) and 2.0 g/l of A-Lube (a lubricant). The fibrillation-inducing step was then run on the closed machine with the rope of fabric being transported through the treatment liquid and being subjected to the mechanical action exerted by the air jet. During this step, the treatment liquid was heated up to a temperature of 130°C at a rate of 2°C per minute and held at that temperature for 45 minutes at above atmospheric pressure. The treatment liquid was then cooled down to 50°C before being run out of the machine.

The fabric rope was then given a series of washes in the machine:-
two successive water rinses at a temperature of 40°C for 15 minutes each; then a neutralizing wash in an aqueous bath comprising 2g/l soda ash, 2g/l A-Lube and 2g/l Sandoclean SPJ (detergent) at a temperature of 30°C for 30 minutes; followed by two further water rinses as specified.

The treated fabric rope was then dyed in the same machine using a hot exhaust migration method carried out over a period of 6 hours using an aqueous dyebath containing;-

Procion Navy H-EXL dye at 3 per cent owf (on weight of fabric) (Procion is a trademark of Dystar AG)
Sodium sulphate at 60g/l

Soda ash at 20 g/l
A-Lube at 2 per cent owf (on weight of fabric).

After dyeing and washing off, the fabric was treated with a soft-finish bath. This was an aqueous bath comprising 0.5 g/l of Hansasoft 2707 (a microsilicone softener), 1 g/l Edunine CSA (a cationic softener) and 1 g/l acetic acid (40%). Softening was carried out at a temperature of 40°C for 20 minutes.

In all the wet processing steps described above in this Example, the rope speed used in the modified Thies Rototumbler was 400 metres per minute.

The soft-finish bath was then withdrawn and the machine was run dry firstly to dry the fabric and then to subject it to a dry tumbling or beating action under the impetus of the air jet. The drying was done by raising the air temperature gradually to 140°C whilst monitoring the moisture content of the air. When the fabric was dry, the machine was run dry for 20 minutes at an air temperature of 120°C followed by a further 15 minutes at an air temperature of 70°C, with a rope speed throughout the dry running of 900 metres per minute.

At the end of the process, the fabric rope was removed from the machine and spread out to full fabric width, and the surface of the fabric was examined. It was evenly dyed to a full navy shade, had a uniformly clean surface appearance and had the desired handle and look of a soft-touch surface finish, which was of sufficient development to be characterized as a peach-touch finish.

Example 2

The procedure of Example 1 was repeated using a lyocell fabric comprising a woven fabric of basis weight 200 gsm constructed in a 2 by 1 twill weave from yarn of count

1/20s Ne. The yarn comprised 60 per cent by weight of TENCEL lyocell fibres and 40 per cent by weight of cotton fibres.

The resulting dyed fabric was evenly dyed to a full navy shade, had a uniformly clean surface appearance and had the desired handle and look of a soft-touch surface finish, which was sufficiently developed to be characterised as a peach-touch finish.

Example3

A lyocell fabric comprised a knitted fabric of basis weight 240 gsm knitted in an interlock construction from yarn of count 1/40s Ne comprising 100 per cent TENCEL lyocell fibres.

The fabric was formed into a rope and was treated by the processes described in Example 1 but with the difference that there was no need to carry out a scouring operation. The resulting fabric was evenly dyed, had a uniformly clean surface appearance and had the desired handle and look of a soft-touch surface finish.

Examples 4, 5 and 6

The procedure of Example 3 was repeated using three different knitted fabrics:-

Example 4 - a single jersey construction of basis weight 170 gsm knitted from yarn of count 1/30s Ne comprising 97.5 per cent by weight of TENCEL lyocell fibres and 2.5 per cent by weight of LYCRA elastomeric filament (LYCRA is a trademark of Du Pont Corp.).

Example 5 - a pique construction of basis weight 200 gsm knitted from yarn of count 1/14s Ne comprising 100 per cent TENCEL lyocell fibres.

Example 6 - a Ponti de Roma construction of basis weight 230 gsm knitted from yarn of count 1/14s Ne comprising 100 per cent TENCEL lyocell fibres.

All three knitted fabrics were evenly dyed, had a uniformly clean surface appearance and had a soft-touch surface finish.

Example 7

The process of the invention was operated at laboratory scale to examine the possibility of dyeing the polyester component of a polyester/lyocell blend fabric during the fibrillation-inducing step of the process.

The lyocell fabric sample was a woven fabric having warp yarns of count 1/30s Ne comprising 100 per cent TENCEL lyocell fibres and weft yarns of count 1/53s Ne comprising 100 per cent polyester fibres. The overall TENCEL/polyester blend was 70:30 (by weight).

After the fabric sample had been scoured to remove any size, it was processed in a laboratory rotary dyeing machine, a Roaches Rotadyer. The sample was put into a dyeing tube together with an aqueous treatment liquid comprising 6.4 g/l acetic acid, 1.0 ml/l (millilitres per litre) DS-14 (a dispersing agent), and 1.0 per cent owf of Dispersol Navy XF (a disperse dye for dyeing the polyester weft yarn) at a liquor to fabric ratio of 20:1 (by weight). The tube was then loaded into the machine and processed. The temperature of the treatment liquid was set at 50°C for 5 minutes before being raised to 130°C at a rate of 1.5°C per minute. The treatment liquid was held at 130°C for 45 minutes at superatmospheric pressure and then cooled down to 80°C.

The fabric sample was then removed from the dyeing tube,

thoroughly rinsed with water and then returned to the dyeing tube in order to dye the TENCEL fibre component. The aqueous dye liquor used comprised Procion Navy H-EXL at 1 per cent owf, sodium sulphate at 40 g/l, and Ludisol (an oxidizing agent) at 3 g/l. The liquor to fabric ratio was 20:1.

The fabric sample was dyed in the Roaches Rotadyer using a hot exhaust migration method in which the fabric was first set, at a temperature of 50°C for 5 minutes, in the dyebath auxiliaries before the dye itself was added to the liquor. Next the temperature of the dyebath was raised to 95°C at a rate of 1.5°C per minute, and the treatment liquor held at 95°C for 30 minutes and then cooled down to a temperature of 80°C. Sodium carbonate was then added to the dyebath up to a solution concentration of 15 g/l, and the dyebath temperature was held at 80°C for a further 45 minutes.

The dyed fabric sample was removed from the dyeing tube, rinsed with water and then washed in an aqueous solution comprising 2 g/l Sandopur SR (a detergent) for 20 minutes at a temperature of 95°C before being rinsed with water again and then allowed to dry in air.

The dry fabric sample was softened in an aqueous softening bath comprising 2 per cent owf Hansasoft 2707 and 3 g/l Sandacid (buffered acetic acid). The bath was applied to the fabric in a Tupesa garment dyeing machine at a temperature of 40°C for 20 minutes. The fabric was then tumble-dried in a garment tumble-drying machine at a temperature of 80°C for 50 minutes followed by a 20 minute cooling-down period.

The fabric sample was evenly dyed to a navy shade and had a clean, soft-touch finish. The soft-touch finish was not so pronounced as the finish achieved in the commercial scale trials described in the previous Examples but was sufficient to demonstrate the feasibility of combining the

carboxylic acid treatment with the disperse dyeing of the polyester component of the fabric.

Example 8

The procedure of Example 7 was repeated using a woven lyocell fabric having a warp of count 1/30s Ne comprising 65 per cent by weight of TENCEL lyocell fibres and 35 per cent by weight of viscose rayon fibres and a weft of count 1/57s Ne comprising 100 per cent polyester fibres. The overall blend proportions were 46 TENCEL lyocell:25 viscose rayon:29 polyester (by weight).

At the end of the procedure, the lyocell fabric was dyed and finished to the same standard as the fabric processed according to Example 7.

Example 9

A lyocell fabric comprised a knitted fabric of basis weight 240 gsm knitted in an interlock construction from 100 per cent TENCEL lyocell fibres. The fabric was formed into a fabric rope which was loaded into a pilot-scale liquor jet dyeing machine (a Colora machine). The machine was rated for use at above atmospheric pressure.

The treatment liquid for use in the fibrillation-inducing step was then introduced into the machine. It was an aqueous solution comprising 0.8 g/l formic acid (100%) and 4 g/l A-lube (a lubricant). This treatment liquid was set at a temperature of 50°C. The machine was then run in a closed condition, with the rope of fabric being transported through the machine by the jets of treatment liquid. During this step, the treatment liquid was heated up to a temperature of 130°C at a rate of 1.5°C per minute and held at that temperature for 45 minutes at above atmospheric pressure. The treatment liquid was then cooled down to 50°C before being run out of the machine.

The fabric rope was then rinsed, dyed and given a soft finish treatment in the same jet dyeing machine, using the

recipes and conditions described in Example 1.

At the end of this sequence of operations, the fabric rope was removed from the jet dyeing machine and loaded into the modified Thies Rototumbler machine referred to in Example 1 for a dry beating operation as described in Example 1.

After removal from the Rototumbler, the fabric was spread out to full width and examined. It was evenly dyed with a uniformly clean surface appearance and had the desired handle and look of a soft-touch surface.

Claims

1. A process for producing a dyed and finished lyocell fabric having a clean, soft-touch finish, comprising subjecting a lyocell fabric to a wet processing treatment using mechanical action to induce fibrillation on the surface of the fabric and then dyeing the fabric before giving it a tumbling treatment to develop the clean, soft-touch finish, characterised in that the fibrillation-inducing step is carried out at above atmospheric pressure, using as the wet processing liquid an aqueous solution of a carboxylic acid which is at a temperature which is above the boiling point of the solution at atmospheric pressure.

2. A process according to claim 1, characterised in that the lyocell fabric is processed in fabric rope form.

3. A process according to claim 2, characterised in that the fibrillation-inducing step is carried out in a jet dyeing machine.

4. A process according to claim 3, characterised in that the jet dyeing machine is an air-jet dyeing machine.

5. A process according to claim 1, characterised in that the lyocell fabric is in the form of piece goods or a garment.

6. A process according to any preceding claim, characterised in that the carboxylic acid is a lower aliphatic carboxylic acid.

7. A process according to any preceding claim, characterised in that the concentration of the carboxylic acid in the wet processing liquid is in the range from 0.2 to 50 g/l.

8. A process according to any preceding claim, characterised in that the carboxylic acid is acetic acid.

9. A process according to claim 8, characterised in that the concentration of the acetic acid in the wet processing liquid is in the range from 2 to 50 g/l.

10. A process according to claim 9, characterised in that the concentration of the acetic acid in the wet processing liquid is in the from range 3 to 10 g/l.

11. A process according to claim 6, characterised in that the carboxylic acid is formic acid.

12. A process according to claim 11, characterised in that the concentration of the formic acid in the wet processing liquid is in the range from 0.2 to 4 g/l.

13. A process according to claim 12, characterised in that the concentration of the formic acid in the wet processing liquid is in the range from 0.4 to 1.2 g/l.

14. A process according to any preceding claim, characterised in that the fibrillation-inducing step is carried out at a temperature of 120°C to 140°C.

15. A process according to claim 14, characterised in that the time of treatment at the specified temperature range is from 30 to 80 minutes.

16. A process according to any preceding claim, characterised in that the fibrillation-inducing step and the subsequent dyeing step are carried out in the same equipment.

17. A process according to any preceding claim, characterised in that the lyocell fabric comprises a blend

of lyocell fibres and polyester fibres, and in that the polyester fibres are dyed with a disperse dye during the fibrillation-inducing step.

18. A process according to any preceding claim, characterised in that the lyocell fabric is a knitted fabric which is processed in fabric rope form and is also scoured in fabric rope form using the same equipment as is used for the subsequent fibrillation-inducing and dyeing steps.

19. A process according to any preceding claim, characterised in that the tumbling treatment to develop the clean, soft-touch finish is a dry treatment.

20. A process according to claim 19, characterised in that the tumbling treatment is carried out on the lyocell fabric in the form of a fabric rope using a fabric rope tumbling machine.

21. A process according to claim 20, characterised in that the tumbling treatment is carried out using an air temperature of 50°C to 150°C for a period of from 30 to 50 minutes.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB2004/000833

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 D06M13/188 D06M23/00 D06P5/20 D06P3/60

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 D06P D06M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 02/103104 A (TAYLOR JAMES MARTIN ; TENCEL LTD (GB); COLLINS GEOFFREY WILLIAM (GB)) 27 December 2002 (2002-12-27) cited in the application the whole document	1-21
A	WO 99/02767 A (NOVONORDISK AS) 21 January 1999 (1999-01-21) the whole document	1-21
A	SCHULER S: "VEREDLUNG VON TENCEL A 100 UND LYOCELL LF" May 1999 (1999-05), TEXTILVEREDLUNG, THURGAUER TAGBLATT, WEINFELDEN, CH, PAGE(S) 12-14,16 , XP000880448 ISSN: 0040-5310 the whole document	1

-/--

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
28 June 2004	05/07/2004

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Koegler-Hoffmann, S

INTERNATIONAL SEARCH REPORT

ational Application No
/GB2004/000833

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	TAYLOR J M ET AL: "AN INTRODUCTION TO TENCEL PROCESSING" 1 August 1997 (1997-08-01), INTERNATIONAL DYER, TEXTILE BUSINESS PRESS LTD. LONDON, GB, PAGE(S) 14,16-17 , XP000720164 ISSN: 0020-658X the whole document	1

INTERNATIONAL SEARCH REPORT

International Application No
.../GB2004/000833

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 02103104 A	27-12-2002	EP 1404917 A2	07-04-2004
		WO 02103104 A2	27-12-2002
WO 9902767 A	21-01-1999	AU 7908198 A	08-02-1999
		WO 9902767 A1	21-01-1999