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(54) Title: FLAME RETARDANT LYOCCELL ARTICLE DYED TO GIVE GOOD LIGHT AND WASHING FASTNESS

(57) Abstract: This invention relates to lyocell fibres made flame-resistant by incorporating flame-resistant pigments and dyed with dyestuffs giving good light and washing fastness.



Flame Retardant Lyocell article dyed to give good light and washing fastness

This invention relates to lyocell fibres made flame-resistant by incorporating flame-resistant pigments and dyed with dyestuffs giving good light and washing fastness.

Prior Art

Textile materials vary considerably in their ability to resist flame and hence protect underlying materials. Most fabrics made from natural fibres and from synthetic fibres will burn when exposed to flame. The rate of burn and ease of ignition are determined primarily by the chemical nature of the polymer from which the fibre is made and the construction of the fabric. Many polymers, such as cellulose, polyester and nylon will burn readily. The rate of burn is lower the heavier a fabric is. Wool is the most common natural fibre which has flame resistant properties to some degree – heavy weight wool fabrics will not burn readily and have long been used in firefighter's clothing.

Fabrics can be treated to make them flame resistant by applying an appropriate chemical to the fabric. The first FR treated fabrics used inorganic salts such as aluminium hydroxide, antimony trioxide and borates to make cotton fabrics flame resistant. These were effective but were non-durable to washing.

Organic phosphorous containing compounds that are reacted onto the cotton either by grafting or network formation are more durable and are widely used.

Two of the leading brand names are Proban® and Pyrovatex®. While these finishes are durable, they can be removed by harsh chemical treatments and the level of finish reduces with the number of washing cycles. The finish application has an adverse stiffening effect on the fabric. Flame retardant finished cotton fabrics are in use for a wide variety of applications where it is required that the fabric does not ignite. When exposed to flame, fabrics of this

type will not burn, but char and become highly embrittled and may break open leaving the wearer's skin or other underlying materials exposed to the hazard.

The first flame resistant cellulosic man made fibres produced were made by the viscose process. A high viscosity liquid flame resistant additive was dispersed in the spinning solution prior to extrusion of the fibre. The liquid was trapped in the cellulose by physical means as very small bubbles. The result was effective as a flame resistant fibre, but the additive could be removed by repeated washing. The strength of the fibre was reduced in proportion to the amount of additive included. The additive was withdrawn from the market due to safety concerns and production of the fibre was discontinued.

An improved flame resistant viscose fibre can be produced by using a solid pigment flame retardant. Fibre of this type will be referred to as FR viscose. The pigment is finely ground and mixed with the spinning solution prior to extrusion of the fibre. The result is a dispersion of the insoluble particulate additive in the fibre. The strength of the fibre is reduced in proportion to the amount of additive included. All of the cellulose in the fibre contains some of the additive and the additive cannot be removed by washing or normal fabric dyeing or finishing processes. Hence the result of the process is an inherently flame resistant fibre. A well-known fibre of this kind is Visil®, which contains silica pigment flame retardant.

A further improvement can be achieved by incorporating the solid pigment flame retardant in the spinning solution used to produce modal fibre. The modal process is a modified viscose process designed to produce a fibre with a higher strength and higher wet modulus than normal viscose. The resultant fibre containing the flame retardant pigment is inherently flame resistant. It is stronger than fibre produced by the viscose process and gives fabrics with higher strength and better stability. Fibre of this type will be referred to as FR Modal but note that the properties of the fibre do not conform to the BISFA definition of modal fibre. Proven flame retardant pigments for this kind of fibres

are organic phosphorous compounds and a preferred pigment is Exolit® (2'-oxybis[5,5-dimethyl-1,3,2-dioxaphosphorinan]2,2'disulphide).

FR Modal is normally used in blend with other flame resistant fibres to produce fabrics which have a combination of the properties of the fibres in terms of strength and physical performance, aesthetics, comfort and physiological effects on the wearer. FR Modal is only rarely used in 100% form in a few applications in the field of apparel such as metallised fabrics or fabrics which are mixtures of two or more yarns. On its own its performance is inadequate in a number of respects compared to other products.

The most recently introduced manmade cellulosic fibre is lyocell. It is produced by a solvent spinning process. The solvent is an amine oxide, which is non-toxic. A slurry of cellulose in a mixture of amine oxide and water is prepared. Water is removed from the slurry by evaporation and as the water content decreases, the cellulose dissolves in the amine oxide producing a solution which is a viscous liquid above 80°C. The solution is extruded through spinneret holes into a water bath. The solvent is diluted by the water and the cellulose precipitates to form a fibre. In the remainder of the process, the fibre is washed to remove any amine oxide solvent, cut into staple fibre, finished with a lubricant and antistat and then dried.

The amine oxide solvent is recycled in a closed loop in the factory. Recovery rates of greater than 99.5% are achieved. Recycling of the additive means that the effect of the process on the environment is very low. It is also essential for the economics of the process.

Lyocell is much stronger than viscose and is stronger than cotton in both the wet and the dry state. It is used in apparel, home furnishings, workwear and nonwovens. Over 90% of the world's lyocell production is produced by Lenzing AG and branded TENCEL®.

The superior properties of lyocell make it possible to produce a version which contains an incorporated flame retardant and which will have superior properties compared to FR viscose and FR Modal. This version will be named

FR lyocell for the purposes of this invention. FR lyocell will be capable of producing blend fabrics with superior properties to current fabrics and will also be capable of producing 100% FR lyocell fabrics with enhanced comfort and physiological effects compared to existing fabrics. FR lyocell is the subject of patent application WO 2012/083318.

FR lyocell as described in WO 2012/083318 is made using a flame retardant which is a solid condensate prepared from the reactants used in the Proban process. The condensate is finely ground to particles of about 1 micrometer and then mixed into the viscous liquid solution used to produce the lyocell fibre during its preparation. In the dried finished fibre, each particle of the flame retardant is completely surrounded by cellulose. Under normal textile processing and use, it cannot be removed from the fibre because it is insoluble and entrapped within the fibre.

It is necessary for many applications to produce fabrics of an appropriate colour. The colour needs to be stable during processing, use and care of the fabric. In particular the colour needs to be stable to washing and to exposure to light. The colour fastness to washing and the colour fastness to light exposure can be measured using standard test methods such as those listed in BS ISO 105-A01 - Textiles – Tests for Colour Fastness.

It is well known that very stable colouration can be achieved in cellulosic fibres by including a pigment in the fibre during manufacture. This method uses a pigment which is itself stable in the conditions of laundering and when exposed to light. The pigment is physically entrapped within the fibre and cannot be removed. The result is a stable colouration for the life of the fibre under normal conditions of use.

However, this approach is not appropriate in many cases. The decision on the colour of a garment has to be made during fibre manufacture. Only a limited range of colours can be made available due to the high costs of changing from one colour to another and the high minimum batch sizes that can be produced.

Dyeing fibre, yarn or fabric is the approach more commonly used for colouration of cellulosic fibres. Non-FR cellulosic fibres can be dyed using a wide range of reactive dyes which are fixed to the cellulose by covalent bonds between the dye molecules and the cellulose molecules. Dyes are available which give good wash fastness and good light fastness and hence the colour of the cellulose fibre is stable during washing and during exposure to light.

Cellulose fibres, yarns or fabrics can also be dyed to stable colours using vat or sulphur dyes. Vat dyes are insoluble substances used for cotton dyeing. They usually contain keto groups, $C=O$, which are reduced to $C-OH$ groups, rendering the dye soluble (the leuco form of the dye). The dye is applied in this form, then oxidized by air or oxidizing agents to precipitate the pigment in the fibres. Indigo and anthroquinone dyes are examples of vat dyes. Sulphur dyes are dyes applied by this technique using sodium sulphide solution to reduce and dissolve the dye. Sulphur dyes are used for cellulose fibres. As both dye types produce a fast color by impregnating fiber with a reduced soluble form that is then oxidized to an insoluble form, they shall jointly be called "re-oxidized dyes" for the purposes of this invention. These dyes are fixed in the fibre by a reaction which creates a solid pigment particle within the structure of the cellulose in the fibre. These pigment particles are physically entrapped within the fibre and cannot be removed by laundering. Vat and sulphur dyes have good light stability. Hence cellulosic fibres, yarns or fabrics dyed with these dyes have good colour stability during laundering and exposure to light.

Problem

During early experiments with dyeing of FR lyocell fabrics it was found that when a fabric was dyed with reactive dyes, the light fastness and washing fastness were significantly worse than for a non-FR lyocell fabric. Because the dye molecules do not achieve their normally stable state when cross-linked to the cellulose the colour they provide is less stable to light. As a result FR lyocell fabric dyed with reactive dyes has poor light stability and poor stability to washing.

It has also been found that application of direct and reactive colours will not meet the fastness requirements for either conventional apparel or protective clothing owing to a reduction in light fastness of the dyes when compared with the same dyestuffs applied to non-FR lyocell fibres. The ionic bonded dye
5 molecules are easily removed by washing giving reduced wash fastness. The light fastness is also reduced.

The objective of this invention is to produce a dyed FR lyocell fabric suitable for use for protective clothing and similar applications and which is washable in industrial laundries with excellent dye fastness and resistance to highly
10 aggressive laundry conditions and a process for producing it.

Description

It is an object of the present invention to provide a dyed FR lyocell fibre which is characterised by being dyed using a dye of the re-oxidized type or an acid
15 dye and by a colour fastness to light of higher than 5 and colour fastness to washing of higher than 4, both according to ISO 105.

For the purposes of this invention the term "fibre" always includes staple fibres as well as – wherever applicable – continuous filaments. Therefore the dyed FR lyocell fibre according to the invention preferably is a staple fibre or a
20 continuous filament fibre.

The group of dyes of the re-oxidized type contains the vat dyes and the sulphur dyes. The group of acid dyes contains the pure acid dyes and the pre-metallised acid dyes.

In a preferred embodiment the light fastness and fastness to washing the
25 dyed FR lyocell fibre are both greater than 5 according to ISO 105.

As described above, the FR additive used to produce FR lyocell as described in WO 2012/083318 is an oxidised condensate of a tetrakis hydroxyalkyl phosphonium salt with ammonia and/or a nitrogenous compound which

contains one or several amine groups. The product is alkaline and contains cationic groups.

The FR lyocell fibres produced can be dyed and spun into yarns, or undyed fibres can be spun into yarns and subsequently dyed or furthermore undyed yarns can be made into fabric by conventional knitting or weaving processes before being dyed in fabric form.

Therefore in a preferred embodiment the dyed FR lyocell fibre is part of a yarn. The fibre can be dyed as a part of the yarn prior to production of a fabric. In case that the fibre is a continuous filament this includes the dyeing of the filament yarn by package dyeing or hank dyeing. Continuous filaments after spinning, coagulation and washing are already obtained in the form of filament bundles which are called filament yarns.

In a preferred embodiment the dyed FR lyocell fibre is part of a fabric. The fibre can be dyed as a part of the fabric.

In order to achieve the necessary fastness and retain the inherent light fastness of the applied dyestuff, the fibre, yarn or fabric should preferably be dyed with dyestuffs of the re-oxidized type, i. e. vat or sulphur. Examples of such dyes are the Indanthren range of vat dyes and Cassulfon range of sulphur dyes from DyStar.

The dye molecules of these types of dye react to form insoluble pigment particles within the cellulose structure. It was found that this is unaffected by the presence of the FR additive.

The re-oxidized dyes are applied to the fibre, yarn or fabric in a reduced or leuco form. The leuco form is created by adding alkali and reducing agent into the dye bath. At the end of the dyeing cycle, the original insoluble form can be reformed within the fibre structure by carrying out an oxidising treatment using hydrogen peroxide. This pigment form is trapped within the fibre structure and is unaffected by the fibre additive giving fastnesses that are characteristic of these dyestuff classes.

FR lyocell can also be dyed with dyes normally used for colouration of nylon or wool. Dyestuffs of the acid type, which can be either of the simple acid or pre-metallised acid type, can successfully be applied to FR lyocell and give good fastness properties. It is believed that the cationic groups on the FR additive act as dye sites for these types of dye.

Acid dyeing on nylon or wool is a simple dyeing process very similar to that of direct dyeing on cellulose. No complicated fixation procedures are needed and the dyeing method is very rapid.

Direct dyes could be applied to the fibre of the invention, but we have seen light fastness to be adversely affected as with reactive dyes also.

The recommended dyes are primarily but not exclusively the re-oxidized dyes, i.e. vat and sulphur dyes and the dyes can be applied using batchwise, semi continuous and continuous application methods.

The process for producing the product of the invention includes the dyeing of the fibre to the desired colour by using dyeing processes of the re-oxidized or the acid types, i. e. the types vat, sulphur, acid or pre-metallised acid. The dyeing of the fibre may be done at any stage; fibre, yarn, fabric or garment.

Therefore in a preferred embodiment the dyed FR lyocell fibre is a staple fibre dyed by stock dyeing.

The FR lyocell fibre may be dyed on-line during fibre production. For example during continuous filament spinning, the filaments could be passed through a dye bath to pick up the dyestuff of the type vat or sulphur in the leuco form and then passed through a reaction chamber where the yarn is exposed to hydrogen peroxide which oxidises the leuco form of the dyestuff back to the insoluble form inside the structure of the fibre. A similar process could be used to produce dyed staple fibre by applying the dye to a washed tow prior to cutting or to a bed of wet cut fibre after cutting and then oxidising it back to the insoluble form inside the fibre.

Therefore in another preferred embodiment the dyed FR lyocell fibre is dyed by on-line dyeing during fibre production.

Table 1 summarises the light fastness results from a series of trials, some of which are described in more detail in the examples below. The results demonstrate that whereas FR viscose (in Table 1 referred to as "Lenzing FR") and non-FR lyocell (in Table 1 referred to as "Tencel cl.") can be dyed to good fastness using any of the methods of dyeing normally used for cellulosic fibres, FR lyocell (in Table 1 referred to as "Lyocell FR") was only be dyed to good fastness by using re-oxidizing dyeing, i. e. sulphur dyeing or vat dyeing.

Table 1

Fiber Type	Dyestuff	Type	Light fastness
Lyocell FR	2,5% Remazol blue R spez.	reactive	1.5
Lenzing FR	2,5% Remazol blue R spez.	reactive	7
TENCEL cl.	2,5% Remazol blue R spez.	reactive	7
Lyocell FR	1% Sirius Rubin K-2BL	direct	3
Lenzing FR	1% Sirius Rubin K-2BL	direct	7
TENCEL cl.	1% Sirius Rubin K-2BL	direct	7
Lyocell FR	14% Hydrosol black	sulfur	7
Lenzing FR	14% Hydrosol black	sulfur	7
TENCEL cl.	14% Hydrosol black	sulfur	7
Lyocell FR	4% Indanthren DB	vat	7
Lenzing FR	4% Indanthren DB	vat	7
TENCEL cl.	4% Indanthren DB	vat	7
Lyocell FR	1% Procion blueHEXL	reactive	3
Lenzing FR	1% Procion blueHEXL	reactive	5.5
TENCEL cl.	1% Procion blueHEXL	reactive	5.5
Lyocell FR	4,2% Novacron	reactive	3
Lenzing FR	4,2% Novacron	reactive	5
TENCEL cl.	4,2% Novacron	reactive	5

A product of this invention is a fabric made entirely of FR lyocell or containing a proportion of FR lyocell which is of a colour suitable for the intended application and has light and washing fastness properties which are suitable for the application. The fabric of the invention may be woven, knitted or nonwoven. Any available process may be used to make the nonwoven fabric including air laying, wet laying, spun laying, needling of a carded or air laid

web, hydroentanglement or any other method of producing a nonwoven fabric. Spun or continuous filament yarns made in whole or in part from FR lyocell or any other fibre type may be included in a nonwoven or may form a significant part of it.

- 5 The FR lyocell fibres can be used to produce a nonwoven fabric from the undyed fibre and then the fabric can be dyed. The FR lyocell fibres can also be dyed and then used to produce a nonwoven fabric.

Therefore a further object of the invention is a dyed FR lyocell fibre which is part of a nonwoven fabric, wherein the fabric is a nonwoven fabric produced
10 by air-laying, carding and needling, hydroentangling, spun laying or any other nonwoven fabric manufacturing method.

The FR lyocell fibre may be blended with other FR fibres such as meta-aramid, para-aramid, modacrylic, PBI and any other FR fibre and may be blended with other non-FR fibres including cotton, wool, silk, linen, lyocell,
15 modal, viscose, nylon, acrylic or polyester.

Therefore a further object of the invention is a dyed FR lyocell fibre which is part of a yarn and wherein the yarn contains, besides the dyed FR lyocell fibre, one or more other fibre types taken from the group containing meta-aramid, para-aramid, modacrylic, PBI, nylon, wool, cotton, silk, linen, FR
20 modal, FR viscose, non-FR lyocell, modal and viscose.

Therefore yet a further object of the invention is a dyed FR lyocell fibre which is part of a fabric and wherein the fabric contains, besides the dyed FR lyocell fibre, one or more other fibre types taken from the group containing meta-aramid, para-aramid, modacrylic, PBI, nylon, wool, cotton, silk, linen, FR
25 modal, FR viscose, non-FR lyocell, modal and viscose.

Each blend must be tested to ensure that it is capable of producing a fabric with the required flammability performance for the intended application.

A further object of the invention is the use of the fibre as described above for the manufacture of a yarn. Therein the dyeing of the fibre according to the invention can be done in the fibre stage as well as in the yarn stage.

Yet a further object of the invention is the use of the fibre as described above
5 for the manufacture of a fabric. Therein the dyeing of the fibre according to the invention can be done in the fibre stage as well as in the yarn stage or in the fabric stage.

Garments made from the fabric are also products of the invention. The product of the invention may be used for the production of all types of
10 garments worn where accidental exposure to flames is a possibility. It can be used for jackets, coats, trousers, boilersuits, coveralls, shirts, sweaters and jumpers, sweatshirts, T-shirts, children's nightwear, adults nightwear, socks, aprons, gloves and gauntlets, hoods for head protection other headwear and any other garment that may be worn when accidental exposure to flame or
15 other source of ignition is a possibility.

Other articles made from the fabric are also products of the invention. They include articles which may be accidentally exposed to flame or other source of ignition such as shoe and boot components, welding screens, fire curtains, tents, upholstery, home furnishings including curtains, sleeping bags,
20 tarpaulins and any other similar articles made in whole or in part from fabric. They also include articles made from nonwoven fabric including filters, interlinings, coating substrates, wipes, cleaning cloths, disposable or short-life clothing, padding and barrier layers for upholstered furniture and other applications where there is a risk of ignition from accidental exposure to flame
25 or other source of ignition.

Therefore a further object of the invention is the use of the fibre as described above for the manufacture of a textile article including garments worn where accidental exposure to flames or other sources of ignition is a possibility such as jackets, coats, trousers, boiler suits, coveralls, shirts, sweaters and
30 jumpers, sweatshirts, T-shirts, children's nightwear, adults nightwear, socks, aprons, gloves and gauntlets, hoods for head protection, other headwear and

any other garment that may be worn when accidental exposure to flame or other source of ignition is a possibility and other articles made from the fabric which may be accidentally exposed to flame or other source of ignition including shoe and boot components, welding screens, fire curtains, tents, upholstery, home furnishings including curtains, sleeping bags, tarpaulins, filters, interlinings, coating substrates, wipes, cleaning cloths, disposable or short-life clothing, padding and barrier layers for upholstered furniture and any other similar articles made in whole or in part from fabric. Therein the dyeing according to the invention is possible in any previous stage as well as in the garment stage.

Another object of the invention is a textile article made using the fibre as described above including garments worn where accidental exposure to flames or other sources of ignition is a possibility such as jackets, coats, trousers, boiler suits, coveralls, shirts, sweaters and jumpers, sweatshirts, T-shirts, children's nightwear, adults nightwear, socks, aprons, gloves and gauntlets, hoods for head protection, other headwear and any other garment that may be worn when accidental exposure to flame or other source of ignition is a possibility and other articles made from the fabric which may be accidentally exposed to flame or other source of ignition including shoe and boot components, welding screens, fire curtains, tents, upholstery, home furnishings including curtains, sleeping bags, tarpaulins, filters, interlinings, coating substrates, wipes, cleaning cloths, disposable or short-life clothing, padding and barrier layers for upholstered furniture and any other similar articles made in whole or in part from fabric.

The invention will now be illustrated by examples. These examples are not limiting the scope of the invention in any way.

Examples

Assessment of colour fastness

In the following examples, the colour fastness to washing and the colour fastness to light exposure are measured. This is done using standard test

methods such as those listed in BS ISO 105-A01 - Textiles – Tests for Colour Fastness. The principle of these tests is that a sample of fabric is washed using a standard washing process or exposed to light as described in the standard. The treated fabric is then compared to an untreated sample of fabric and the difference in colour is compared using a grey scale. The higher the number (maximum is 7) assigned to the colour fastness the better it is. Thus in the case of wash fastness, a low figure would indicate a fabric with poor colour fastness which undergoes a significant change in colour when washed. A high figure would indicate that the change in colour on washing is very small or even no change at all. In the test method used the maximum colour fastness which can be assessed is 7.

Example 1

A 1/30s Ne yarn made from FR lyocell fibres manufactured as described in Patent Application WO 2012/083318 was wound onto dyer's packages with a density of 350g/l or 25 Shore density. In an enclosed yarn package dyeing machine the yarn was dyed with a vat dye using a procedure as recommended by the dyestuff manufacturer. The process was as follows:-

A dye bath was prepared consisting of the following ingredients in water: 4% Indanthren Dark Blue DB, 2g/l Setamol WS (Protective colloid and dispersing agent from BASF), 0.3g/l Albigen A (levelling agent from BASF), 4ml/l NaOH (50%), 5g/l Sodium Hydrosulphite, 20g/l Glaubers Salt. The yarn packages were dyed at 60°C for 75 minutes. After dyeing the yarn was washed and oxidized as follows: The yarn packages were washed in the dyeing machine with cold water. The yarn was then oxidised using a bath consisting of the following ingredients: 3ml/l Hydrogen Peroxide (30%), 2ml/l Acetic Acid (80%),

The yarn was oxidised in the dyeing machines for 30 minutes at 60°C. The yarn packages in the dyeing machine were then rinsed with hot water and then with cold water and then soaped off with a bath consisting of: 2g/l Kieralon JET (wetting agent / detergent from BASF), 2g/l Soda Ash. The soaping off was done for 30 minutes at 95°C. The yarn packages were then rinsed with hot water and then with cold water. The yarn packages were then

unloaded from the dyeing machine and dried by carousel drying in a conventional manner. The resultant yarns were then re-wound from the dyers packages onto weavers cones before weaving into a 2x1 twill fabric of 200gsm weight.

- 5 The fabric was assessed for colour fastness to washing and to light. The resultant fabric gave excellent fastness to washing and light. The light fastness according to ISO 105-B02 – Colour Fastness to Artificial Light - was 7. Wash fastness according to ISO 105-C06 - Colour Fastness to Domestic and Commercial Laundering was 4-5, change of shade and staining of
10 adjacent cloths.

These results are as would be expected on a lyocell fibre without the FR pigment additive.

Example 2

- A woven 2/1 twill fabric (200gm-2) made from the same undyed yarns from
15 example 1 was prepared in open width by scouring with a non-ionic detergent and sodium carbonate at 90°C, then dried on a stenter. The fabric was then dyed using Indanthren vat dyes in a fully continuous pad, dry, pad steam process as recommended by DyStar: Pad the fabric in a bath consisting of the following ingredients in water: 30g/l Indanthren Red FBB, 1g/l Sera Quest C-
20 PX (sequestering agent from DyStar), 10g/l Sera Gal M-IP (migration inhibiting agent from DyStar), 2g/l Sera Wet C-UD (wetting agent from DyStar).

- The fabric was skied (left open to the air) for 20 seconds before predrying in an infrared drier to 25-40% residual moisture, before completing the drying
25 process in a hot flue at 120°C for 45-100 seconds. The fabric was then padded at 80% wet pick up with a bath consisting of the following ingredients in water: 2g/l Sera Sperse C-SN (dispersing agent from DyStar), 2g/l Sera Sperse MI-S (dispersing agent from DyStar), 100g/l NaOH (38°Be), 50g/l Sodium Hydrogen sulphite. The fabric was then steamed for 60 seconds at

102°C in a tight strand steamer before being continuously washed in an eight box system. The boxes contained:

Boxes 1 & 2. Water at 40°C

Box 3. 3ml Hydrogen peroxide (35%) in water at 60°C

5 Box 4. 3ml/l Hydrogen Peroxide (35%) and .5ml/l Acetic Acid (80%) in water at 60°C

Boxes 5 & 6. 2g/l detergent and 1g/l Soda Ash in water at 95°C

Box 7. Water at 80°C

Box 8. Water at 40°C

10 After washing, the fabric was dried on the stenter. The resultant fabric gave excellent fastness to washing and light. The light fastness according to ISO 105-B02 was 6. Wash fastness according to ISO 105-C06 was 4-5, change of shade and staining of adjacent cloths.

15 These results are also as would be expected on a lyocell fibre without the FR pigment additive.

Example 3

The undyed woven fabric from example 2 above was dyed using Cassulfon Carbon CMR, a sulphur dye from DyStar on a jet dyeing machine. The fabric was loaded into the jet dyeing machine with the machine manufacturer's
20 recommended amount of water at 40°C. The following chemicals were added: 14% Cassulfon Carbon CMR liquid, 16.5% Sulphydrate F 150 (reducing system from DyStar), 4g/l Soda Ash, 2g/l Stabilisal S liq (Reducing agent stabiliser from DyStar), 1g/l Primasol NF (wetter from DyStar).

25 The dyeing bath temperature was increased to 95°C over 20 minutes and then 20g/l common salt was added. The dyeing was continued for 45 minutes before cooling the bath to 70°C. The liquid was then dropped to drain. The fabric was then washed off in the jet dyeing machine as follows: The fabric was rinsed with warm water at 40°C and then rinsed with cold water. 1%

Hydrogen Peroxide (35%) and 1% Soda Ash were added to the water in the machine and circulated with the fabric for 10 minutes at 40°C. The fabric was rinsed with warm water at 40°C and then rinsed with cold water. The fabric was then stenter dried.

- 5 The black fabric was assessed for colour fastness to washing and to light. The resultant fabric gave excellent fastness to washing and light. The light fastness according to ISO 105-B02 was 6. Wash fastness according to ISO 105-C06 was 4-5, change of shade and staining of adjacent cloths.

These results are also as would be expected on a lyocell fibre without the FR
10 pigment additive.

Example 4

- The undyed fabric produced in example 2 was dyed using a pre-metallised dye as follows: The fabric was loaded into a jet dyeing machine, filled with water to the normal dyeing level giving a normal 10:1 liquor to goods ratio.
- 15 The temperature was raised to 40°C and the following additions made: 2% Isolac Blue S-RL (pre-metallised acid dye from DyStar), 2g/l Glauber's salt, 1g/l Biavin TCC (lubricant from CHT). The pH was adjusted to 6.5 using acetic acid (80%). The dye bath temperature was increased to 95°C at 2°C / minute and dyeing continued for 60 minutes. The fabric was then washed with water
- 20 at 40°C and then washed with cold water until the washings were clear of unfixed dyestuff. The fabric was dyed to a dark navy shade of good wash and light fastness.

A sample of standard lyocell (i. e. without FR pigment additive) included in the same dyebath at the same time was only stained a pale blue at the end of the
25 dyeing process.

Example 5

The undyed fabric from example 2 was dyed with a reactive dye to demonstrate the poor performance achieved using a conventional cellulosic dyeing method. The fabric was loaded into a jet dyeing machine, filled with

water to the normal dyeing level giving a normal 10:1 liquor to goods ratio.

The temperature was raised to 40°C and the following additions were made:

60g/l Common Salt, 1g/l Ludigol (anti-reduction additive from BASF), 1g/l

Albegal FFA (non-foaming wetting agent from Huntsman), 0.3cc/l Biavin 109

5 (lubricant from CHT). After 10 minutes running, the dyestuff was added over

10 minutes 1% Procion Blue H-EXL (reactive dye from DyStar). The

temperature of the dyeing bath was raised at 2°C/minute to 80°C and dyeing

continued for 30 minutes. 20g/l Soda ash was then added in two equal

portions over 20 minutes and dyeing continued for 60 minutes. After which

10 time the bath was cooled to 60°C and the exhausted dyebath dropped to drain.

The hydrolysed dye and chemicals were then washed clear of the fabric by

repeated washing as follows: Wash hot and cold. Neutralise with 1g/l Acetic

acid (60%). 10 minutes at 40°C. Wash off at 95°C for 20 minutes in 1g/l

15 Kieralon JET. Complete washing by rinsing hot (60°C) and cold. The fabric

was then stenter dried. The blue fabric was assessed for colour fastness to

light and washing. The resultant fabric gave poor fastness to washing. The

light fastness according to ISO 105-B02 was 3.

This result is significantly lower than would be expected for a fabric made

20 using a lyocell fibre without the pigment additive, where a figure of 5-6 would be achieved.

Claims

1. Dyed FR lyocell fibre which is characterised by being dyed using a dye of the re-oxidized type or the acid type and by a colour fastness to light of higher than 5 and colour fastness to washing of higher than 4, both according to ISO 105.
2. Dyed FR lyocell fibre according to claim 1, wherein the fibre is a staple fibre or a continuous filament fibre.
3. Dyed FR lyocell fibre according to claim 1, wherein the fibre is a staple fibre dyed by stock dyeing.
4. Dyed FR lyocell fibre according to claim 1, wherein the fibre is dyed by on-line dyeing during fibre production.
5. Dyed FR lyocell fibre according to claim 1, wherein the fibre is part of a yarn.
6. Dyed FR lyocell fibre according to claim 5, wherein the fibre is dyed while being part of the yarn prior to production of a fabric.
7. Dyed FR lyocell fibre according to claim 5, wherein the yarn contains, besides the dyed FR lyocell fibre, one or more other fibre types taken from the group containing meta-aramid, para-aramid, modacrylic, PBI, nylon, wool, cotton, silk, linen, FR modal, FR viscose, non-FR lyocell, modal and viscose.
8. Dyed FR lyocell fibre according to claim 1, wherein the fibre is part of a fabric.
9. Dyed FR lyocell fibre according to claim 8, wherein the fibre is dyed while being part of the fabric.
10. Dyed FR lyocell fibre according to claim 8, wherein the fabric contains, besides the dyed FR lyocell fibre, one or more other fibre types taken from the group containing meta-aramid, para-aramid, modacrylic, PBI, nylon, wool, cotton, silk, linen, FR modal, FR viscose, non-FR lyocell, modal and viscose.
11. Dyed FR lyocell fibre according to claim 8, wherein the fabric is a nonwoven fabric produced by air-laying, carding and needling, hydroentangling, spun laying or any other nonwoven fabric manufacturing method.

12. Dyed FR lyocell fibre according to any of claims 5 or 8, wherein the light fastness and fastness to washing are both greater than 5 according to ISO 105.
13. Use of the fibre according to any of claims 1 or 5 for the manufacture of a yarn.
14. Use of the fibre according to any of claims 1, 5 or 8 for the manufacture of a fabric.
15. Use of the fibre according to any of claims 1, 5 or 8 for the manufacture of a textile article including garments worn where accidental exposure to flames or other sources of ignition is a possibility such as jackets, coats, trousers, boiler suits, coveralls, shirts, sweaters and jumpers, sweatshirts, T-shirts, children's nightwear, adults nightwear, socks, aprons, gloves and gauntlets, hoods for head protection other headwear and any other garment that may be worn when accidental exposure to flame or other source of ignition is a possibility and other articles made from the fabric which may be accidentally exposed to flame or other source of ignition including shoe and boot components, welding screens, fire curtains, tents, upholstery, home furnishings including curtains, sleeping bags, tarpaulins, filters, interlinings, coating substrates, wipes, cleaning cloths, disposable or short-life clothing, padding and barrier layers for upholstered furniture and any other similar articles made in whole or in part from fabric.
16. Textile article made using the fibre according to any of the preceding claims including garments worn where accidental exposure to flames or other sources of ignition is a possibility such as jackets, coats, trousers, boiler suits, coveralls, shirts, sweaters and jumpers, sweatshirts, T-shirts, children's nightwear, adults nightwear, socks, aprons, gloves and gauntlets, hoods for head protection other headwear and any other garment that may be worn when accidental exposure to flame or other source of ignition is a possibility and other articles made from the fabric which may be accidentally exposed to flame or other source of ignition including shoe and boot components, welding screens, fire curtains, tents, upholstery, home furnishings including curtains, sleeping bags, tarpaulins, filters, interlinings, coating substrates, wipes, cleaning cloths,

disposable or short-life clothing, padding and barrier layers for upholstered furniture and any other similar articles made in whole or in part from fabric.