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(54) **Title**: SHRINK RESIST AND INDUSTRIALLY LAUNDERABLE FABRICS MADE FROM VISCOSE, LYOCELL OR MODAL FIBERS CONTAINING PARTICULATE ADDITIVES

(57) **Abstract**: The invention claims a fabric made in whole or in part from a man-made cellulosic fibre which contains a particulate material, which exhibits shrinkage of less than 2.5% in woven form and 5% in knitted form when subjected to 25 cycles of aqueous laundering, wherein the fabric is unresinated and is provided with a silicone elastomer. Additionally a process for the manufacture of such a fabric is claimed.

# SHRINK RESIST AND INDUSTRIALLY LAUNDERABLE FABRICS MADE FROM VISCOSE, LYOCELL OR MODAL FIBERS CONTAINING PARTICULATE ADDITIVES

## Background

Spun dyed viscose and inherently FR viscose are manmade cellulosic fibers that contain finely divided solid particulate materials that perform the required function. In the case of spundyed viscose the particulate material is a pigment of the colour required in the final fibre. In FR viscose the particulate material is a flame retardant in solid form.

Generally, for both fibre types, the particulate material is dispersed in the cellulose solution before the fibre is formed. The particulate material may be added to the cellulose solution at any point before spinning. For example it may be added with the wood pulp which is the source of the cellulose or I may be added during dissolution of the cellulose xanthogenate to form the spinning solution or it may be added to a cellulose solution during mixing or immediately prior to extrusion as a slurry.

Among other things, the advantages of spin dyeing are as follows:
Large batches having the same shade of colour may be obtained.
The dyeing exhibits excellent fastness to wetting.
The conventional dyeing process is unnecessary, resulting in savings of energy and raw materials (chemicals and water) and reduced risk of environmental pollution by waste water.
There is no loss of dye.
The problem of unevenness or streaking during dyeing does not occur.
A homogeneous distribution of the dye in the fibre and thus uniform dye penetration are achieved.

For FR viscose an organophosphoric flame retardant additive is mixed as an aqueous predispersion into a solution of cellulose xanthogenate, filtered and spun into a regenerating and coagulation spin bath.

An example of spun dyed viscose products is Viscose Color from Lenzing AG. Lenzing FR is an inherently FR cellulosic fiber, that has a higher tenacity and wet modulus than conventional viscose.
An example of a conventional inherently FR viscose is So FR viscose fiber from Waxman Speciality Products.

Shrinkage is a very serious problem when aqueous systems are applied to viscose containing fabrics and because of this shrinkage, viscose containing fabrics generally require dry cleaning as opposed to washing in aqueous media. This is a disadvantage for viscose and it would be a significant advance if viscose containing fabrics could be treated so as to make them washable in water.

When the fabric is made from 100% viscose and subjected to aqueous washing, shrinkage may be as high as 18-20%. The shrinkage problem is also severe with a polyester / viscose fabric, because the viscose shrinks so much, and the polyester so little, that the fabric becomes distorted due to the difference in the rate of shrinkage of the two fibers.

Spun dyed viscose and FR viscose fabrics exhibit this problem in the same way as fabrics made from normal viscose fibre.

The conventional approach to solving this fabric shrinkage problem is to apply a finish to the fabric. Methods have been devised for treating cellulosic fiber containing products which impart durable crease retention, wrinkle resistance and smooth drying characteristics to these products and at the same time reduce the shrinkage significantly. These methods use resins or pre-condensates of the urea-formaldehyde or substituted urea- formaldehyde such as dimethylol-di-hydroxy-ethylene-urea (DMDHEU) types to produce a resin treated durable press product. However, viscose fabrics finished with these systems generally lose significant strength and their useable life is reduced.

Summary of the Invention

It is possible to achieve good shrinkage performance, durable press properties and good strength retention in a fabric which contains viscose fiber with a process that utilises acid catalysts and high concentrations of formaldehyde. Such a process in disclosed in United States Patent 6,511,928 The process produces consistent results. The resulting fabric may be cleaned using normal washing detergents and water and does not require the fabric to be dry cleaned.

The solution to the problem described above is a fabric made in whole or in part from a man-made cellulosic fibre which contains a particulate material, which exhibits shrinkage of less than 2.5% in woven form and 5% in knitted form when subjected to 25 cycles of aqueous laundering, wherein the fabric is unresinated and is provided with a silicone elastomer.

In a preferred embodiment of the invention the man-made cellulosic fibre is a viscose or modal fibre.

In another preferred embodiment of the invention the man-made cellulosic fibre is a lyocell fibre.

Preferably the particulate material is a colored pigment.

In another preferred embodiment of the invention the particulate material is a flame retardant.

Preferably the man-made cellulosic fibre which contains a particulate material is blended or comixed with one or more other fibres such as but not limited to polyester, nylon, modacrylic, para-aramid, meta-aramid wool or cotton

Preferably the fabric is treated with a composition comprising formaldehyde and catalyst. More preferably the composition comprises from about 5.18% to about 7.4% by weight, formaldehyde.

In a preferred embodiment of the invention the composition consists essentially of water, formaldehyde, silicone elastomer and catalyst.

Also a subject matter of the invention is a process for treating a fabric comprising pigment containing viscose or modal fibres or blends containing such, wherein the fabric is treated by the process comprising steps of:

- contacting the fabric with a liquid composition comprising catalyst and from about 5.18% to 7.4% formaldehyde by weight,
- heat curing the fabric and
- cleaning the fabric after heat curing,
- wherein the fabric is provided with a silicone elastomer and is unresinated.

Preferably prior to the step of heat curing, the fabric has a moisture content of more than 20% by weight.

Preferably the liquid composition further comprises an amount of silicone elastomer composition sufficient to provide from 0.1% to 5% on weight of fabric, solids.

Preferably the liquid composition further comprises a dimethyl silicone elastomer emulsion.

Preferably the liquid composition comprises the dimethyl silicone elastomer emulsion at a level sufficient to provide from 0.053% to 2.65% on weight of fabric, silicone solids.

Preferably the composition comprises from about 14% to about 20% by weight, formalin, and wherein the formalin comprises 37% by weight, formaldehyde.

A silicone elastomer may also be included with the formaldehyde and acid catalyst as part of the finishing process to help achieve the required fabric hand. The result is, viscose containing fabrics having unique properties including low shrinkage which makes them water washable and which

provides a distinct commercial advantage over viscose containing fabrics produced using conventional resination.

Treating fabrics made from viscose fibre which contains a particulate pigment and/or a particulate flame retardant with the high level formaldehyde system results in performance levels that are unachievable by any other technology. The fabrics that result have excellent hand, do not suffer from significant loss of strength, have good easy care and wrinkle resistance and do not have excessive shrinkage levels.

Spun Dyed Viscose and Formaldehyde Finishing shows:

- Excellent wet fastness than cannot be equalled by conventional dyeing technology
- Lowest impact to the environment – conventional dyeing is unnecessary which results in significant savings in water and chemicals.
- Washing shrinkage of typically less than 3% by area, even in knitted constructions.
- Excellent durable press ratings typically better than 3.5 that can be classified as non-iron.
- Resistance to industrial laundry processes giving suitability for demanding workwear applications.

Inherently FR viscose and Formaldehyde Finishing shows:

- Washing shrinkage of typically less than 3% by area, even in knitted constructions.
- Excellent durable press ratings typically better than 3.5 that can be classified as non-iron.
- Resistance to industrial laundry processes giving suitability for demanding workwear applications.
- Retention of FR performance through extended washing cycles.

**Examples**

Example 1

Viscose Color spun dyed black fiber was spun into 1/30s Ne yarn. From this yarn single jersey fabrics were knitted both in 100% form (150gsm) and 95/5 blend with Lycra (212gsm).

The fabric was finished using the formaldehyde system:

- Pad in
  - 10% Formaldehyde
  - 3% Freecat LF (from Freedom Chemicals) – based on Magnesium Chloride / Citric Acid
  - 1.5% Silicone elastomeric softener
  - Wet pick up 90%
- Dry and Cure 10 minutes at 150°C
- Wash off to remove excess formaldehyde in hot water and neutralise in a dilute solution of sodium bicarbonate.

The shrinkage results for the untreated fabric and the formaldehyde treated fabric are given in Table 1:

Table 1:

| Sample | Shrinkage 1 wash (40°C) | Shrinkage 25 washes |
|---|---|---|
| 100% Viscose untreated | -21% length, +8% width | -28%, +14% |
| 95/5 Viscose / Lycra untreated | -9.75%, +2% | -13%, +2.5% |
| | | |
| 100% Viscose treated | -3%, -4.25% | -3%, -5.5% |
| 95/5 Viscose / Lycra treated | -0.75%, -1% | -1.5%, -1.75% |

The shrinkage results for the untreated fabric are unacceptable for any normal application. The shrinkage results for the treated fabric are excellent and would be acceptable for any normal application.

Example 2

Viscose Color spun dyed black fiber was spun into 1/20s Ne yarn, in both 100% and as a 50/50 intimate blend with spun dyed black polyester. Each of the two yarns was then woven into a 3x1 twill fabric. Both fabrics had a 50/50 polyester / viscose warp 36ends / cm.

Fabric A also had a 50/50 polyester / viscose weft (32 picks / cm)
Fabric B had a 100% viscose weft also with 32 picks / cm.

The fabrics were split into halves:
Half was treated with conventional resin:

- 60g/l Knittex FEL
- 15g/l Magnesium Chloride
- 20g/l Perisoft GS (silicone softener)
- 2g/l Kieralon JET (wetting agent)

- Pad (80% wet pick up), dry at 120°C, cure 45 secs at 175°C

The second half was treated with formaldehyde as in example 1.

Shrinkage tests gave the results shown in Table 2:

Table 2:

| Sample | Shrinkage 1 wash 60°C with tumble dry | Shrinkage 10 washes 60°C with tumble dry |
|---|---|---|
| Fabric A resin | -2%, -2.7% (wp, wt) | -3.7%, -4.4% |
| Fabric B resin | -3%, -4% | -4.7%, -6.6% |
| Fabric A HCHO | -0.3%, 0 | -0.3%, -0.3% |
| Fabric B HCHO | -1%, -1% | -1.3%, -1.6% |

The fabrics treated with formaldehyde gave shrinkage results that are comparable with the best normally achieved in any textile processing. The fabrics treated with conventional resin gave results that indicate significant shrinkage making them unsuitable for some applications. The fabrics were then tested for a range of properties appropriate for an industrial laundry application:

Example 3

A 100% Lenzing FR knitted single jersey fabric was treated in formaldehyde as in example 1. Test results were as shown in Table 3:

Table 3:

| Sample | Bursting Strength (lbs) | Wash shrinkage (1 40C wash and tumble dry) | Wash shrinkage (25 40C washes with intermediate tumble drying) |
|---|---|---|---|
| Untreated | 87 | -15% length, +1% width | -21%, +5% |
| HCHO Finished | 62 | -1.5%, -0.5% | -3.75%, 0% |

The shrinkage results for the untreated fabric are unacceptable for any normal application. The shrinkage results for the treated fabric are excellent and would be acceptable for any normal application.

Example 4

A 2x1 twill weave fabric of Lenzing FR in blend with Kermel (aramid fiber): 30/70 Lenzing LF / Kermel, 230gsm, was treated in formaldehyde as in example 1.

Test results were as shown in Table 4:

Table 4:

| Sample | Tensile Strength (lbs) | Tear Strength (lbs) | Wash shrinkage (1 40C wash and tumble dry) | Wash shrinkage (25 40C washes with intermediate tumble drying) | DP Rating |
|---|---|---|---|---|---|
| Untreated | 164wp, 150 wt | 7.9wp, 9.9wt | -0.25wp, -1.25wt | -2.5%, -3.25% | 3.5 |
| HCHO Finished | 151wp, 113 wt | 7.9wp, 6.3wt | 0%, 0% | -0.75%, -0.25% | 3.75 |

The fabric treated with formaldehyde gave a significant improvement in the shrinkage results compared with the untreated fabric and gave a slight improvement in the durable press rating. The effect on tensile strength and tear strength was less than would be expected from conventional resin finishing.

Claims:

1. A fabric made in whole or in part from a man-made cellulosic fibre which contains a particulate material, which exhibits shrinkage of less than 2.5% in woven form and 5% in knitted form when subjected to 25 cycles of aqueous laundering, wherein the fabric is unresinated and is provided with a silicone elastomer.

2. A fabric according to claim 1, wherein the man-made cellulosic fibre is a viscose or modal fibre.

3. A fabric according to claim 1, wherein the man-made cellulosic fibre is a lyocell fibre.

4. A fabric according to claim 1, wherein the particulate material is a colored pigment.

5. A fabric according to claim 1, wherein the particulate material is a flame retardant.

6. A fabric according to claim 1 wherein the man-made cellulosic fibre which contains a particulate material is blended or comixed with one or more other fibres such as but not limited to polyester, nylon, modacrylic, para-aramid, meta-aramid wool or cotton

7. A fabric according to claim 1, wherein the fabric is treated with a composition comprising formaldehyde and catalyst.

8. A fabric according to claim 2, wherein the composition comprises from about 5.18% to about 7.4% by weight, formaldehyde.

9. A fabric according to claim 2, wherein the composition consists essentially of water, formaldehyde, silicone elastomer and catalyst.

10. A fabric according to claim 7, wherein the liquid composition further comprises an amount of silicone elastomer composition sufficient to provide from 0.1% to 5% on weight of fabric, solids.

11. A fabric according to claim 7, wherein the liquid composition further comprises a dimethyl silicone elastomer emulsion.

12. A fabric according to claim 11, wherein the liquid composition comprises the dimethyl silicone elastomer emulsion at a level sufficient to provide from 0.053% to 2.65% on weight of fabric, silicone solids.

13. A fabric according to claim 7, wherein the composition comprises from about 14% to about 20% by weight, formalin, and wherein the formalin comprises 37% by weight, formaldehyde.

14. A process for treating a fabric comprising pigment containing viscose or modal fibres or blends containing such, wherein the fabric is treated by the process comprising steps of:

a. contacting the fabric with a liquid composition comprising catalyst and from about 5.18% to 7.4% formaldehyde by weight,

b. heat curing the fabric and

c. cleaning the fabric after heat curing,

wherein the fabric is provided with a silicone elastomer and is unresinated.

15. A process according to claim 14, wherein prior to the step of heat curing, the fabric has a moisture content of more than 20% by weight.

# INTERNATIONAL SEARCH REPORT

International application No
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A. CLASSIFICATION OF SUBJECT MATTER
INV. D06M13/127 D06M15/643 D06M15/647
ADD.

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)
D06M D01F

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 practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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| Date of the actual completion of the international search | Date of mailing of the international search report |
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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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Information on patent family members

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