

1

2

3,477,806

TREATMENT OF CELLULOSE TRIACETATE WITH SELECTED COMPOUNDS TO INTRODUCE STRETCH PROPERTIES

William C. Sturkey, Charlotte, N.C., assignor to Celanese Corporation, a corporation of Delaware

No Drawing. Filed Oct. 15, 1965, Ser. No. 496,666

Int. Cl. D06m 5/14

U.S. Cl. 8—131

6 Claims

ABSTRACT OF THE DISCLOSURE

Textile materials having improved stretch properties are produced by treating textile material comprising cellulose organic acid esters having less than 0.29 free hydroxyl groups per anhydroglucose unit with a compound selected from the group consisting of beta-butoxyethyl acetate, acetyl triethyl citrate and benzyl alcohol.

This invention relates to textile materials having stretch properties. It particularly relates to a process for imparting stretch properties to textile materials composed of cellulose triacetate and to the novel product made therefrom. It specifically relates to a process for introducing stretch properties into cellulose triacetate by solely chemical means.

Textile materials composed of cellulose organic esters of low, free hydroxyl content have many desirable properties. Thus, after such materials have been properly heat treated they may be ironed at high temperatures, are fairly resistant to glaze or shrinkage on pressing with moist steam, and they generally have excellent crease resistance, dimensional stability, and wash-fastness. Even though these properties have enabled textile materials of this type to receive wide acceptance in the market place it has heretofore not been possible for these materials to find an end use where stretch fabrics are either required or desired, since such materials normally only have from 1% to 5% stretch.

In order to create a stretch fabric out of textile materials of cellulose organic acid esters of low, free hydroxyl content, it previously has been necessary to blend other filamentary materials having stretch properties into the fabric, e.g., by incorporating into the fabric yarns composed of rubber or elastic synthetic polyurethane. There has not been in the prior art any means taught for imparting true recoverable stretch properties to textile materials composed of cellulose organic acid esters as the stretch component.

As shown in U.S. Patent No. 2,900,669 to Max H. Booth, when materials composed of cellulose acetate are immersed in suitable warm, aqueous swelling agents, these materials extend in length and in diameter without the application of tension or external stress. This elongation through the use of swelling agents is substantially retained after cooling and drying. In other words, the extension of the textile material under these conditions is basically irreversible.

However, it has been discovered that if a select group of treating agents are used under usual conditions this extension could be reversed in that the cellulose triacetate in the textile material is shrunk and the shrinkage is made relatively permanent or becomes "set." Therefore, fabrics made in accordance with this invention may be stretched at least 16% and in many cases may be stretched in excess of 40% and upon relaxation of the tension producing the stretching will substantially return to its original orientation thereby having a permanent growth of no more than 5%.

Accordingly, it is an object of this invention to teach a method for imparting stretch properties to textile mate-

rials. It is another object of this invention to produce a novel textile material having stretch characteristics. It is still another object of this invention to produce a fabric composed of cellulose triacetate having stretch properties in an extremely facile and economical manner. It is a particular object of this invention to produce a textile material composed of cellulose triacetate having at least 16% stretch and no more than 5% permanent growth by contacting such material with a select group of treating agents under usual conditions.

Therefore, in accordance with one embodiment of this invention, a textile material composed of cellulose organic acid esters of low, free hydroxyl content having unusually high stretch properties can be produced by a method which comprises treating such material with a compound selected from the group consisting of β -butoxyethyl acetate, acetyl triethyl citrate, and benzyl alcohol under conditions whereby said material is shrunk an amount sufficient to produce at least 16% stretch and no more than 5% permanent growth in said textile material.

As used in the description of this invention and in the claims to the invention the terms "cellulose organic acid esters of low, free hydroxyl content" refer to cellulose esters having less than 0.29 free hydroxyl groups per anhydroglucose unit, e.g., cellulose acetate having an acetyl value of at least 60%, preferably at least 61% calculated as combined acetic acid hereinafter referred to as cellulose triacetate. Other lower aliphatic acid esters of cellulose of low hydroxyl content also may be employed in this invention. For example, the propionate, butyrate, mixed acetate-propionate, mixed acetate-butyrate, and mixed acetate-formate esters of cellulose may be used with satisfactory results. It is distinctly preferred, however, that cellulose triacetate be used as the textile material having stretch properties imparted thereto in the practice of this invention.

The contacting of the textile material may be carried out in any manner known to those in the art as long as the required amount of treating agent contacts the textile material as set forth in greater detail hereinbelow. It is preferred, however, that the contacting be carried out in an aqueous liquor having contained therein the β -butoxyethyl acetate, acetyl triethyl citrate, or benzyl alcohol. The apparatus used in the practice of this invention may be pressure equipment or preferably can be carried out in a conventional winch or jig. The contacting may be performed at temperatures in excess of 50° C. up to temperatures beyond which the filamentary material is damaged and preferably is carried out at temperatures ranging from 60° C. to 95° C., a contact time or residence time of from 5 to 120 minutes, preferably 30 to 90 minutes; and under conditions substantially free of tension thereby allowing the filamentary material in a transverse direction to shrink. Liquor ratio may range from 2:1 to 100:1, preferably, from about 3:1 to 8:1 in the case of the jig; and from about 10:1 to 50:1 in the case of winch contacting. Since the liquor ratio will govern the proportion of treating agent in contact with the fiber at any given concentration of the agent in the liquor, it is apparent that the optimum concentration will to some extent be related to the liquor ratio. In all cases, however, the apparatus and liquor ratios should be chosen so that an agent dosage of from 35% to 300% by weight, based on the weight of cellulose triacetate in the textile material, will be used for the contacting medium, and preferably from 40% to 150%.

The cellulose triacetate textile material used in accordance with this invention may be in the form of staple fibers, tow, continuous filament or staple fiber yarns, fabrics and/or finished articles. They may be used alone or in admixture with other fibrous materials such as

wool, cotton, rayon, silk, nylon, glass, polymers and/or copolymers of vinylidene compounds such as acrylonitrile, ethylene, propylene, vinyl chloride, vinyl acetate, vinylidene cyanide, and the like. Preferably the textile material is a woven fabric composed of cotton warp and cellulose triacetate filling wherein the cellulose triacetate in the filling may be either alone or in admixture with these other fibrous materials; but, preferably comprises a major proportion of the filling. Additionally, the cotton yarn used in the warp should not be finer than a forty (40) singles count but may be as coarse as desired. The filamentary material used in the filling should have a denier from 75 to 600 or the equivalent, with a preferred denier ranging from 200 to 450. The filamentary material may have a slight twist, e.g., from 0.5 to 5 turns per inch, imparted thereto prior to treatment although this is not necessary.

It has also been found that stretch characteristics vary with fabric construction. Thus, greater stretch may be imparted to a cellulose triacetate fabric having a relatively loose plain weave construction than with a relatively firm or tightly woven fabric such as blousewear or rainwear. Thus, the construction of the fabric must be such that the filamentary material in the direction desired for stretch is free to move and thereby shrink under the treatment of the agent. Accordingly, a plain weave fabric having approximately equal thread spacing in the warp and filling are preferred since this construction permits a high sinusoidal yarn crimp to be imparted to the filling threads by the action of the agent in causing the filling yarn to shrink against the resistance of the warp threads.

Prior to contacting the textile material with the desired treating agent as shown herein the material may be scoured, desized, bleached and/or treated in any of the other conventional operations. Following such contact the treated material can also be finished in usual fashion by rinsing, extracting, loop drying (temperature 225°–300° F. for 3–10 minutes, preferably from 4–6 minutes), and the like. However, in all of these pretreatment or post-treatment operations the fabric should not be subjected to more than minimum amount of tension in the direction that it is desired to impart stretch until the fabric has been finally finished as desired by those skilled in the art, e.g., after heat-setting.

In a further embodiment of this invention, the textile material which has been in contact with the treating agent under the proper conditions as set forth hereinabove is further subjected to a heat treatment, preferably under minimum tension in the transverse direction, at a temperature of from 375° F. to 425° F. In a preferred practice of this latter embodiment, the stretch property has been imparted to the filling and the heat treatment is carried out under minimum warp tension. As used herein, minimum warp tension is defined as that tension which will not introduce more than 10% extension of the warp. Preferably this tension should impart no more than 5% extension to the warp.

The heat treatment can be effected by contact as with metal cans or rolls, by hot air as on an enclosed frame, or by radiant heat. In each situation, however, this heat treatment should be carried out such that the textile material is subjected to a minimum amount of tension in the direction that has been treated to impart stretch. Preferably, the heating is carried out by contact with a metal can.

The textile materials produced by the practice of this process are novel, per se, in that they are composed of

cellulose triacetate having at least 16% stretch and no more than 5% permanent growth. Specifically, these novel fabrics contain at least a major proportion of cellulose triacetate in the filling and preferably at least one other filamentary material in the warp; said fabric having from 16% to 40% stretch and from 0.5 to 5% permanent growth. The novel textile material, as a new composition of matter, is characterized by the fact that the cellulose triacetate portion of said material has been swollen by the action of the agent as evidenced, usually by a crystal order index (COI) in excess of 1.00 after being treated with the treating agent in accordance with the process of this invention in contrast with, usually, a crystal order index of less than 1.00 for the untreated cellulosic triacetate. The most important change, however, in the fiber is its swollen state and not necessarily a crystal order index change even though such a COI change usually accompanies the swelling action.

In order to determine this change in crystal structure the yarn can be subjected to X-ray diffractometry. The degree to which treating agent action on the fiber has altered the crystal structure can be measured by observing the number and/or the sharpness of the major diffraction arcs of the fiber. Thus, for example, cellulose triacetate has four pronounced diffraction peaks at 8, 10, 12.6 and 16° 2 θ where 2 θ is the angle which the diffracted X-ray beam makes with respect to the incoming beam. Depending upon the degree of treating action these peaks show differences in intensity, height and weight. A quantitative representation of these differences is found by measuring the ratio of peak height to weight at half height for a series of diffraction peaks from X-ray diffractometer curves or microphotometered X-ray photographs. One specific measure of the crystalline structure is the "crystalline order index." It represents the average of the ratios of each peak height to its weight at half height of the four diffraction peaks at 8, 10, 12.6 and 16° 2 θ .

Therefore, the crystalline structure developed in the yarn from the agent treatment should be such that its crystalline order index measured as described above is at least 1.00 and preferably 1.2 to 1.5. An X-ray photograph of such a yarn would show four weak but distinct diffraction arcs at 8, 10, 12.6 and 16° 2 θ in the case of cellulose triacetate, while the original untreated yarn would show only two diffuse broad arcs or diffraction rings, thus indicating a crystal order index of less than 1.00.

The following examples further illustrate the invention:

EXAMPLE I

Two types of woven fabrics, both having the filling composed of 100% cellulose triacetate (CTA), were scoured, desized and bleached in a conventional manner and then were contacted with a treating agent under the following conditions:

Apparatus: winch.

Agent: β -butoxyethyl acetate (BCA) in water (concentration=13 grams BCA/liter water).

Agent Dosage: 40% by weight based on the weight of cellulose triacetate.

Liquor ratio: 30:1 (grams of agent to grams of cellulose triacetate).

Time: 60 minutes.

Temperature of Liquor: 80° C.

After usual rinsing, extracting, and loop drying (temperature of from 225°–300° F. for 3 to 10 minutes), the following results were obtained:

Warp Yarn	Filling Yarn	Type	Thread Count		Stretch, percent	Permanent Growth, percent
			Warp	Filling		
(a) 20/1 Cotton.....	200/2z/52 CTA..	Denim..	64	58	25.3	4.2
(b) 20/1 Cotton.....	300/2z/80 CTA..	Denim..	64	42	16.3	3.9

5

EXAMPLE II

The greige fabric 20/1 cotton: 200/2 Z/52 CTA from Example I was treated under the same conditions except that 75% aqueous BCA (75 grams BCA/liter water) and then 150% aqueous BCA (50 grams BCA/liter water), respectively, was used with the following results:

	75% BCA (1)	150% BCA (1)
Stretch, percent.....	26.5	23.9
Permanent Growth, percent.....	2.9	2.6

¹ Based on the weight of cellulose triacetate in fabric.

EXAMPLE III

The fabrics of Example I were treated again as in Example I except that, additionally, following contact with the treating agent, the fabrics were finally heat treated at 410° F. over Morrison cans under minimum warp tension¹ with the following results:

Type:	Stretch, percent	Permanent Growth, percent
(a).....	26.6	1.4
(b).....	22.5	2.2

EXAMPLE IV

A greige fabric composed of 18/1 spun cellulose triacetate in the warp and 200/34 false twist textured cellulose triacetate in the filling were subjected to scouring, desizing and bleaching; and then treated with various treating agents under the following condition:

Apparatus: Winch.

Agent dosage: 40% by weight based on weight of cellulose triacetate (13 grams BCA/liter water).

Liquor ratio: 30:1.

Temperature: 90° C.

Time: 90 minutes.

Heat treatment: Morrison cans @ 410° F. under minimum warp tension.¹

The resulting fabrics had the following properties:

Treating Agent	Stretch, percent	Permanent Growth, percent
Acetyl triethyl citrate.....	¹ >15.0	¹ <5.0
Benzyl alcohol.....	39.1	2.9
β -butoxyethyl acetate.....	16.0	2.7

¹ Estimated.

EXAMPLE V

The fabric of Example IV was processed as in Example IV except that different treating agents were used, to wit: β -methoxyethyl acetate, glycol diacetate, and triacetin (glycerol triacetate). In each run the stretch property was less than 10% so that these agents are deemed ineffective in producing stretch into cellulose triacetate fabrics and filamentary materials.

Even though this invention has been described with reference to a liquor bath having water as the carrier it is to be understood that other carriers may be used with equal success. For example, ethylene glycol may be used or other polyhydric alcohols such as diethylene glycol. In fact, any carrier for the treating agent which will have no significant effect on the characteristics of the textile material being treated can be used to practice this invention.

The treating agent may be added to the carrier in any manner known to those skilled in the art. It can be added to an agitated vessel having the carrier therein. In the case of BCA, for example, the BCA is added to the water with high agitation since this treating agent has low solubility in water. Preferably, a commercial dispersing agent is

¹ Sufficient tension to cause approximately 5% extension of the warp.

6

used to aid in producing the liquor of proper concentration.

As used herein, "stretch" is defined with reference to how much extension may be imparted to the textile material and have it returned spontaneously to its original orientation. As used herein, the term "permanent growth" relates to the degree to which the textile material fails to return to its original orientation after relaxation of the tension which was used to impart the stretch. The procedure used for measuring stretch and percent growth is well known to those skilled in the art. However, it basically consists of using a stretch tester consisting of a vertical frame with sets of fixed clamps at the top and a means of attaching a weight to the bottom of the test specimen. A gauge is attached to measure the extension of the fabric. A holding mechanism is provided to keep the specimen extended a predetermined amount.

The stretch test is performed at 70° F., 65% relative humidity using a 2½" x 14½" strip, with the longer length parallel to the stretch direction. The test strip is also raveled to 2" in width. A four pound weight is used to stretch the test strip and the specimen is cycled three times between zero and four pounds at approximately five seconds per cycle. The stretch is measured at the bottom of the fourth cycle and converted into percent (%) stretch.

For measuring percent permanent growth, place the specimen in the apparatus and stretch the specimen a predetermined amount; to wit: 15% for filling stretch measurement and 25% for a warp stretch measurement; and hold in this position for a minimum of sixteen (16) hours. After this time has elapsed, the specimen is released and allowed to slowly return to its normal position. The remaining stretch under slack conditions is measured and converted to percent (%) permanent growth.

If it is desired to impart warp stretch characteristics to the fabric composed of cellulose triacetate, such fabric can be placed in a suitable tenter frame which fixes the filling dimensions and the fabric is fed into the liquor with sufficient overfeed to allow the warp to shrink.

Even though this invention has been described as applicable to woven fabrics it is also contemplated that this invention can be practiced on knitted fabrics such as warp knits and circular knits.

Having described my invention, what I desire to secure by Letters Patent is:

1. A method for introducing stretch properties into a textile fabric wherein the filamentary material in the direction desired for stretch is free to move, said fabric consisting essentially of cellulose esters of lower alkanolic acids, said esters having less than 0.29 free hydroxyl groups per anhydroglucose unit, which comprises treating said fabric with from 35 percent to 300 percent, based on the weight of said cellulose ester, of a compound selected from the group consisting of beta-butoxyethyl acetate, acetyl triethyl citrate and benzyl alcohol at a temperature of from 60 degrees to 95 degrees centigrade for a period of time of from 5 to 120 minutes and under conditions substantially free of tension, allowing the fibers to shrink in a traverse direction, producing a fabric having from about 16 percent to about 40 percent stretch, based on the original length of the fabric, and having no more than 5 percent permanent growth in said textile fabric, based on the original length of the fabric.

2. A method according to claim 1 wherein said compound is beta-butoxyethyl acetate.

3. A method according to claim 2 wherein said textile fibers are formed into a woven fabric and a major proportion of the filling consists essentially of cellulose triacetate.

4. A method of producing a woven fabric having a stretch of from about 16 percent to about 40 percent, based on the original length of said fabric, which comprises contacting a woven fabric containing a major proportion of cellulose triacetate in the filling, said filling filamentary material having a denier of from 75 to 600,

7

with from about 40 percent to 150 percent by weight, based on the weight of cellulose triacetate, of a compound selected from the group consisting of beta-butoxyethyl acetate, acetyl triethyl citrate and benzyl alcohol in an aqueous liquor, at a temperature of from about 60 degrees centigrade to 95 degrees centigrade, a liquor ratio of from about 2:1 to 100:1, for a period of from about 5 to 120 minutes, and under conditions substantially free of tension in the filling, permitting said fabric to shrink in a traverse direction and producing a woven fabric having from about 16 percent to about 40 percent stretch, based on the original length of the fabric, and having no more than 5 percent permanent growth in the filling, based on the original length of the fabric.

5 5. A method according to claim 4 wherein said woven fabric contains spun yarns in the warp consisting essentially of cotton, and wherein said fabric is contacted with beta-butoxyethyl acetate.

10 6. A method according to claim 4 wherein said fabric is removed from contact with said liquor and further subjected to heat setting under minimum warp tension

8

in the presence of a heat source maintained at a temperature of from about 375 degrees Fahrenheit to 425 degrees Fahrenheit.

References Cited

UNITED STATES PATENTS

3,025,128	3/1962	Finlayson et al.	8—131
3,132,919	5/1964	Bull et al.	8—131
3,135,576	6/1964	Browne et al.	8—131
3,153,563	10/1964	Warner et al.	8—131 XR
3,167,385	1/1965	Mann et al.	8—131

FOREIGN PATENTS

795,821 5/1958 Great Britain.

GEORGE F. LESMES, Primary Examiner

J. P. BRAMMER, Assistant Examiner

U.S. Cl. X.R.

8—114.5, 132; 28—76; 264—342