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CELLULOSE ESTER TEXTILE MATERIALS

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1 Claim. (Cl. 106—181)

ABSTRACT OF THE DISCLOSURE

Cellulose acetates, both secondary and triacetates, are rendered more resistant to delustring by incorporating in textile articles made from them 0.1 to 5% (on the weight of the cellulose acetate) of orthophthalic, isophthalic, terephthalic, trimesic or adipic acid. The articles can be made by dry spinning solutions of the cellulose acetate containing the additive.

This application is a continuation-in-part of 416,148 filed Dec. 4, 1964, now abandoned.

This invention relates to textile materials comprising fibres of cellulose acetates having improved resistance to delustring.

It is well known that fibres of cellulose acetates tend to become delustred, i.e. to acquire a more or less dull or opalescent appearance, when they are treated in aqueous baths at high temperatures.

The conditions under which this effect starts to manifest itself vary in accordance with the nature and origin of the acetate employed, and notably in accordance with its degree of acetylation. In fibres of secondary cellulose acetates, the effect begins to appear generally between 80° and 85° C., while in fibres of the so-called cellulose triacetates (i.e. cellulose acetates having a combined acetic acid content above 59%), the effect appears only at much higher temperatures, which may even be above 100° C. in some cases.

Various means are known for avoiding or lessening the delustring of cellulose acetate fibres in aqueous baths at high temperatures, for example the addition of protective salts to the said baths, or replacement of the cations retained as impurities in the cellulose acetate from its production.

It has now been discovered that fibres of cellulose acetates sensitive to delustring in aqueous baths at high temperatures may be improved in this respect without modifying the cellulose acetate spun or the spinning operation, by adding to the spinning solution a small proportion of certain acids.

Accordingly in one aspect this invention consists in textile articles comprising threads or fibres of cellulose esters and containing between 0.1% and 5% by weight of at least one acid of the general formula $R(COOH)_n$, in which R is a hydrocarbyl radical of 4 to 6 carbon atoms, and n is 2 or 3.

Also in accordance with the invention threads and fibres of cellulose acetates which are unusually resistant to delustring are made by extruding in the form of fibres a solution of a cellulose acetate containing 0.1–5%, of the weight of the cellulose acetate, of an acid of formula $R(COOH)_n$, in which R is a hydrocarbyl radical of 4–6 carbon atoms and n is an integer from 2 to 3, and setting the fibres so formed by evaporation of the solvent (dry spinning).

The term "cellulose acetates" is used to denote both so-called secondary cellulose acetates having a combined acetic acid content between 54.5% and 55%, and the product commonly called cellulose triacetate, having a combined acetic acid content between 59% and 61.5%.

Among the acids of formula $R(COOH)_n$, with which good results have been obtained, there may be mentioned orthophthalic acid, isophthalic acid, terephthalic acid, adipic acid and trimesic acid. In some cases, these products may be introduced in the form of their anhydrides, the moisture present in the spinning solution generally being sufficient to convert them to the acids.

The extent to which the delustring threshold temperature is raised varies with the acetate employed and the spinning process.

In the case of dry spin secondary acetate fibres, it is possible to raise the delustring threshold temperature by from 2° to 5° C., depending upon circumstances; although this increase is small in absolute terms, it is of great technological importance, since, as is well known, secondary cellulose acetates commence to dye effectively at about 80° C., and even a slight increase in the margin of safety from the viewpoint of delustring not only makes it possible to improving the depth and levelness of the dyeing, but also provides an important increase in the safety factor with regard to accidental damage due to delustring effect.

In the case of dry spun fibres of cellulose triacetates, the increase in the temperature at which delustring begins may in some cases be as much as 30° C.; this is clearly important, especially in the case of articles which start to become delustred at about 100–105° C. and which may, by means of the present invention, be treated without delustring at temperatures of about 130° C., and possibly higher, so enabling these articles to be subjected to the thermal treatments necessary for producing good noncreasing properties, as well as to be dyed under optimum conditions.

It is also found that different acids exercise their greatest effect, and are preferably employed in association with, either cellulose triacetates or secondary cellulose acetates but not both. Orthophthalic acid is an exception, being very effective, in raising the delustring threshold temperature of both types of cellulose acetate, and is indeed particularly preferred for use in accordance with the invention. On the other hand, the terephthalic acid and trimesic acid, for example, are most effective with secondary cellulose acetates, while isophthalic acid and adipic acids are most effective with cellulose triacetates. The type of cellulose acetate in association with which any particular acid is most effective is readily determined by a simple test.

The following examples illustrate without limiting the invention.

EXAMPLE 1

A solution of 21.5% by weight of cellulose triacetate in a mixture of methylene chloride and methanol (90/10

by weight) is dry spun in a known way to give threads which begin to delustre at 100° C. When 0.2% of orthophthalic acid (calculated on the weight of triacetate) is added to the same solution, which is then spun under the same conditions as before, there is obtained a thread which remains shiny when treated with hot water under pressure up to and even above 130° C., a rise in the delustring threshold temperature of at least 30° C.

The threads thus obtained and the control threads are used to make fabrics for blouses. As is usual with triacetate fabrics, these fabrics are dyed in an aqueous bath at 130° C., this temperature sufficing both to give good depth of dyeing and also to render the fabric crease resistant. It is found that the fabric made with the control thread is dull, while that made with the thread containing orthophthalic acid has remained shiny. If it is desired to maintain the fabric made of the control thread in the shiny condition, it is necessary to keep the temperature to a maximum of 100° C., but in this case the intensity of the shade obtained is lower and the fabric is not crease resistant.

The following table shows the serimetric results obtained on the control thread and on the thread containing the orthophthalic acid.

Treatment undergone by the thread	Breaking strength in g./d.		Elongation at rupture	
	Reference product	Addition of orthophthalic acid	Reference product, percent	Addition of orthophthalic acid, percent
Untreated	1.15	1.25	34	36
Steaming 30 min. at 130° C	1.10	1.12	32	34.8
Pressurised water 1 hour at 120° C	1.08	1.11	32.2	32.6
Heat treatment 24 hours at 110° C	1.12	1.17	32.8	37

It will clearly be seen from this table that the serimetic properties are slightly improved by the presence of orthophthalic acid.

The following table, which relates to the action of light, shows comparative tests carried out with and without introduction of 0.2% of orthophthalic acid by exposure to light in an apparatus known under the trade mark Xenotest, for 158 hours.

Treatment undergone by the thread	Breaking strength in g./d.		Elongation at rupture	
	Reference product	Addition of orthophthalic acid	Reference product, percent	Addition of orthophthalic acid, percent
After exposure	1.15	1.25	34	36
After 50 hours	1.00	1.05	29	29.8
After 98 hours	0.90	1.03	26	30.6
After 158 hours	0.72	0.83	13.6	15.4

This table shows that the orthophthalic acid, also affords protection against degradation by light.

EXAMPLES 2 to 6

The same cellulose triacetate spinning solution is employed as in Example 1, and variable quantities of orthophthalic acid are introduced. After dry spinning, the following results were obtained:

TABLE I

Examples	Quantities of acid (percent)	Delustring threshold temperature, ° C.	Appearance of the thread treated with water at 130° C.	Breaking resistance in g./d.
Control	0	100	Opalescent	1.15
2	0.1	>130	Shiny	1.21
3	0.4	>130	----do	1.20
4	0.8	>130	----do	1.16
5	1.6	110	Slightly opalescent.	1.23
6	3.2	110	----do	1.17

EXAMPLES 7 to 12

Into a spinning solution containing 25% by weight of secondary cellulose acetate in acetone were introduced variable quantities of orthophthalic acid. After drying spinning, the following results were obtained:

TABLE II

Examples	Quantities of acid	Delustring threshold temperature, ° C.	Appearance of the thread treated with water at 90° C.	Breaking strength in g./d.
Control	0	82	Opalescent	1.32
7	0.1	85-90	Very slightly opalescent.	1.32
8	0.2	85-90	----do	1.33
9	0.4	85-90	----do	1.37
10	0.8	85-90	----do	1.32
11	1.6	90	Shiny	1.05
12	3.2	90	----do	1.06

EXAMPLE 13

A solution of secondary cellulose acetate in acetone containing 0.2% of terephthalic acid calculated on the weight of the cellulose acetate is dry-spun by known means. A thread is obtained which becomes very slightly opalescent under the action of water at 85–90° C., while a control thread of the same cellulose acetate becomes distinctly opalescent at 82° C.

EXAMPLE 14

An acetone solution of a secondary cellulose acetate containing 0.1% of trimesic acid calculated on the weight of the cellulose acetate, is dry-spun by known means. A thread is obtained which becomes very slightly opalescent under the action of water at 85–90° C., while a control of the same acetate thread becomes distinctly opalescent at 82° C.

EXAMPLE 15

The same cellulose triacetate solution as in Example 1, but containing 0.2% of isophthalic acid on the weight of the cellulose triacetate, is dry-spun by known means. A thread is obtained which becomes slightly opalescent under the action of water at 105° C., while a control thread starts to become opalescent at 100° C.

EXAMPLE 16

The same cellulose triacetate solution as in Example 1, containing 0.1% of adipic acid, is dry-spun by known means. A thread is obtained which is very slightly opalescent from 105–110° C., while a control thread starts to become opalescent at 100° C. These last examples

show that isophthalic and adipic acids, while they are most effective with secondary cellulose acetates, have some effect also with triacetates.

We claim:

1. Textile articles comprising fibres of cellulose triacetate which fibres contain 0.1 to 3.2% by weight of orthophthalic acid.

References Cited

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