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PROCESSING OF CELLULOSE TRIACETATE

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The present invention relates to novel processes for improving the properties of highly esterified cellulose filamentary materials such as cellulose triacetate.

In accordance with the present invention filamentary materials of organic acid esters of cellulose having fewer than about 0.29 free hydroxyl groups per anhydroglucose unit are treated with hot dilute aqueous solutions of lower aliphatic acid esters of alkoxy-lower alkanols, alkoxy alkoxy-lower alkanols or lower alkylene glycols, of triethyl phosphate, triethylene glycol diacetate, cyclic ethylene carbonate or lactonitrile. The solutions are subsequently washed out and the filamentary materials dried. As compared with untreated filamentary materials the treated materials dye more rapidly and evenly, have a higher safe ironing temperature and are stronger after being subjected to heat treatment.

Organic acid esters of cellulose which can be treated in accordance with the present invention include formates, propionates, butyrates, acetate propionates, acetate butyrates, and the like although cellulose acetate, having an acetyl value of at least about 59% and preferably at least about 61% by weight calculated as acetic acid, is preferred. This material is hereinafter referred to as cellulose triacetate and the invention will be further described with reference to this preferred cellulose ester. The filamentary material may be in the form of fibers, tows, yarns, webs, fabrics, or the like, alone or blended with other fibers which are not damaged by the treatment.

Of the enumerated treating agents the lower fatty acid esters, lower alkyl ethers and lower alkoxy-lower alkyl ethers of hydroxy-lower alkyl esters of lower fatty acids, e.g. ethylene glycol diacetate, β-ethoxyethyl acetate and butoxyethoxyethyl acetate give the best results from the points of view of degree of improvement, economy, ease and permissible latitude in treatment, rapidity and low useful concentrations. The concentration of treating agent may vary from as low as about 1 to 50%, by weight. The temperature of treatment may vary from about 50 to 150° C. and preferably about 75 to 150° C., and the time of treatment may last up to about 3 hours although it preferably is complete in about 1 hour or less, e.g. about 6 seconds at 150° C. Generally the time and temperature vary inversely with each other and with concentration. It is an advantage of the invention that even if the concentration, time and temperature are all simultaneously at relatively high values the filamentary materials will not be damaged, i.e. the process is not sensitive to variations within the broad ranges although in the interest of economy obviously it will be desired to employ the shortest possible times and the lowest possible concentrations and temperatures.

The concentration will vary with the temperature and time and with the identity of the treating agents. Thus for β-ethoxyethyl acetate the preferred concentration is 1 to 25%; for ethylene glycol diacetate 2 to 16%; for butoxyethoxyethyl acetate 1 to 6%; for triethyl phosphate 5 to 25%; for triethylene glycol diacetate 5 to 10%; for ethylene carbonate 1 to 15%; for lactonitrile 1 to 25%.

The treatment may be effected by immersion of the filamentary material in the treating solution for the requisite time, by travel of the filamentary material through a treatment bath and then out of the bath with the distance and speed correlated to give the desired treatment time,

by padding, i.e. immersion followed by squeezing, to a predetermined pick-up, by spraying, or the like. The solution may be initially at relatively low temperatures and steam, hot air or hot surfaces can be employed to achieve the desired treatment temperature. The amount of solution contacted with the fabric is at least about 30% of the fabric weight and usually at least 50%. The solution should be employed in amount sufficient to present about 1 to 50% and preferably 5 to 20% of the ester based on the weight of cellulose triacetate.

If dyeing is to be carried out in combination with the novel treatment, the treatment may be effected simultaneously with the dyeing as by adding a dyestuff to the solution or it may be effected subsequent to dyeing but preferably it is effected prior to dyeing and is followed by washing out of the treating solution prior to dyeing. This permits large amounts of the filamentary material to be treated in a single treatment apparatus, with obvious economies, even if portions of the filamentary materials are intended to be dyed different colors. Also the dilute treating solution recovered by washing is uncolored and can easily be concentrated and re-used.

Preferably following, but possibly prior to, the novel treatment the filamentary material is given a heat treatment such as being contacted for about 10 to 60 seconds with hot air at about 200 to 235° C. in a tenter frame or a hot flue, by 3 to 30 seconds of contact with metal surfaces such as hot cans at about 200 to 235° C., or the like. If the filamentary material is to be dyed, the sequence is preferably pretreatment, followed by dyeing, followed by heat treatment. Upon heat treatment, filamentary materials which have been pretreated in accordance with the present invention are stronger than materials identical except for omission of the pretreatment, whether or not dyeing has taken place.

The invention will be further described with reference to the following illustrative examples wherein all proportions are by weight unless otherwise expressed:

Example I

(a) A portion of fabric woven of cellulose acetate staple fiber yarn having an acetyl value of 61.5% calculated as acetic acid is immersed in 55 times its weight of a 5% solution of β-ethoxyethyl acetate at 95° C. and after 300 seconds is rinsed in hot water, rinsed in cold water and dried.

(b) Employing the Scott ravelled strip method (ASTM Method D–76–53 paragraph 3A) for determining tensile strength and the Elmendorf Tear Tester (ASTM Method D–1424–56T) the pretreated fabric of (a) after heat treatment with radiant heat is about 20 to 30% stronger than the untreated fabric after heat treatment.

(c) The starting material and the end product in (a) are held for 30 seconds between two plates heated to various temperatures. At 375° F. the untreated specimen becomes stiff whereas the treated specimen hardly changes in stiffness even at 425° F.

(d) Upon dyeing a piece of the starting material and of the end product of (a) in identical aqueous baths, containing 1% of Colour Index Disperse Blue 27 on the weight of the fiber, for 1 hour at 95° C. the pretreated fabric picks up 2.5 times as much dye as the untreated fabric. The dyeing of the pretreated fabric, which can subsequently be heat treated as in (b) if desired, is characterized by marked evenness and freedom from streaks. This advantage also extends to tricot constructions where dyed fabrics which have not been pretreated are occasionally streaked.

Similar advantages are achieved when ethylene glycol diacetate or butoxyethoxyethyl acetate is substituted for the β-ethoxyethyl acetate.

Example II

The fabric of Example I can be pretreated by immersion in 55 times its weight of a 10% aqueous solution of triethyl phosphate for 300 seconds at about 95° C. After rinsing with water and drying, the fabric can be heat treated as in Example I. If desired, the fabric can be dyed prior to heat treatment.

Example III

Repeating Example II with the substitution of a 10% aqueous solution of triethylene glycol diacetate for the triethyl phosphate produces approximately the same improvements.

Example IV

The process of Example II is repeated except for use of a 10% aqueous solution of ethylene carbonate for the pretreatment.

Example V

A 10% aqueous solution of lactonitrile can be used as the pretreating agent in the process of Example II.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit of my invention.

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

1. The process which comprises immersing a filamentary material comprising a lower alkanoic acid ester of cellulose having fewer than about 0.29 free hydroxyl group per anhydroglucose unit in an aqueous solution of about 1 to 15% by weight concentration of ethylene carbonate for a duration ranging from a time at least sufficient to increase the safe-ironing temperature up to about 1 hour at about 50 to 150° C., the ethylene carbonate being present in about 1 to 50% of the weight of the cellulose ester.

2. The process recited in claim **1**, including the subsequent steps of washing said filamentary material with water and subsequently heat treating to increase the crystallinity of said lower alkanoic acid ester of cellulose.

3. The process which comprises contacting a filamentary material comprising a lower alkanoic acid ester of cellulose having fewer than about 0.29 free hydroxyl groups per anhydroglucose unit with an aqueous solution of about 1 to 25% by weight concentration of lactonitrile

for a duration ranging from a time at least sufficient to increase the safe-ironing temperature up to about 1 hour at about 50 to 150° C., the lactonitrile being present in about 1 to 50% of the weight of the cellulose ester.

4. The process recited in claim **3**, including the subsequent steps of washing said filamentary material with water and subsequently heat treating to increase the crystallinity of said lower alkanoic acid ester of cellulose.

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