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POLYESTER FIBERS RESISTANT TO RESOILING DURING LAUNDERING

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ABSTRACT OF THE DISCLOSURE

Polyester fibers having improved resistance to resoiling during laundering may be prepared by dispersing a fatty acid ester of a poly(ethylene glycol) in said polyester prior to spinning and then spinning said fibers from said polyester.

This application is a continuation-in-part of Kiefer U.S. Serial No. 273,284, filed Apr. 16, 1963, now abandoned.

This invention concerns a method for improving polyester fibers by treating the polyester with polyethers before melt spinning.

Laundering consists essentially of removing soil from the fabric and keeping the soil in suspension so that it can be rinsed away. Normally, soap or a synthetic detergent is used to wet the fabric and emulsify or disperse the soil. Soap is fairly effective in preventing the soil from redepositing on the fabrics in the laundry solution. However, most synthetic detergents do not perform this important function. Therefore, many laundry detergent formulations contain a detergent additive which prevents the redeposition of the soil on the fabric. A common and widely used detergent additive is carboxymethylcellulose which is very effective in preventing soil from redepositing on cotton fabrics, but which has no effect in preventing redeposition of soil on most synthetic fabrics during laundering. Consequentially, white goods of synthetic fabrics often develop a discoloration when laundered with other fabrics carrying a relatively heavy soil load, since the soil is absorbed from the laundry solution by synthetic fabrics. This is particularly true in the case of polyesters which often have a gray appearance due to the accumulation of soil. Attempts have been made to overcome soil redeposition in the laundering of polyesters, but the conventionally used treatments do not absorb the optical brighteners present in most laundry detergent formulations and are not permanent even under ordinary laundering conditions. These conventional treatments often lose their effectiveness after less than 30 cycles in an automatic washing machine. Also, the suggested treatments are not permanent to dry cleaning and are therefore useful only in fabrics which are never exposed to a dry cleaning bath.

I have now found that when polyesters are treated with certain polyethers under controlled conditions, the polyesters are considerably less susceptible to discoloration caused by the absorption or adsorption of soil during laundering.

One object of this invention is to provide a method for treating polyester fibers to make them less susceptible to redeposition of soil during laundering. Another object is to provide polyester fibers and fabrics which are resistant to soil redeposition during laundering. Another

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object is to provide polyester fibers and fabrics which are resistant to soil redeposition even after dry cleaning.

The above objects are attained by treating polyesters and fabrics in such a way as to disperse a polyether in the polyester before spinning and to then melt spin the polyester having the polyether dispersed therein into a fiber.

Various methods may be employed for incorporating the polyether into the polyester material. For instance, the polyether and polyester can be mixed together by dry mixing in appropriate mixing equipment, such as a Banbury mixer, a sigma blade mixer, or in a revolving drum. The polyester can be formed into pellets, rods, powder, etc., and sprayed with liquid polyether before melt spinning.

The polyether can be dissolved in a volatile solvent for the polyether which is a nonsolvent for the polyester. The polyester resin is then slurried in the polyether solvent solution, and the excess solution drained off. The solvent remaining on the polyester is evaporated and the coated polyester resin dried by heat and/or reduced pressure. Suitable solvents for polyether are water; alcohols, such as methanol, ethanol, and the propanols; ketones, such as acetone and ethyl methyl ketone; esters, such as ethyl acetate and methyl acetate; chlorinated hydrocarbons, such as dichloromethane and dichloroethane; and aliphatic and aromatic hydrocarbons, such as benzene, toluene, hexane, etc.

The polyether and the polyester can be mixed together on a hot roll such as is commonly used in mixing plasticizers with thermo-plastic resins. When this method of mixing is used, it is sometimes advantageous to mix a relatively high concentration of the polyether (5–30 percent) into a low molecular weight polyester. This mixture, commonly called a masterbatch, is then mixed with the unmodified high molecular weight polyester to give the desired amount of polyether in the final mixture. The masterbatch technique is only one of the known methods which may be used to melt mix the polyether and polyester in an extruder such as is commonly used in mixing plasticizers with thermoplastic resins.

The above methods of adding the polyether to the polyester are illustrative of the many methods known to the art and are not intended to limit the practice of my invention.

The polyesters with which my invention is concerned are any of the condensation polymers prepared from diols and dibasic acids or their derivatives. Particularly important are poly(ethylene terephthalate) and poly(1,4-cyclohexylenedimethylene terephthalate).

The polyethers which are useful in carrying out my process are fatty acid esters of poly(ethylene glycols) having a molecular weight of at least 2,000. For instance, I may use poly(ethylene glycols) such as those commercially sold by Union Carbide Company under the trade designations Carbowax 2,000, Carbowax 4,000, Carbowax 20M, Polyox WSR-35, Polyox WSR-205, and Polyox WSR-301. If the poly(ethylene glycol) has a molecular weight less than 2,000, the fatty acid ester derived therefrom will be a smaller molecule which tends to come off during the wash cycle, thereby making for a less permanent treatment.

The fatty acid esters encompassed by this invention are those (both substituted and unsubstituted) derived from fatty acids having 10 to 22 carbon atoms. Examples of these acids would include capric, 3-methylcapric, un-

decanoic, lauric, 4-chlorolauric, tridecanoic, myristic, pentadecanoic, 4-ethyl-5-chloropentadecanoic, arachidic, heneicosanoic, behenic, etc., acids. Fatty acids having less than 10 carbon atoms do not produce the desired results, while those having more than 22 carbon atoms offer no additional advantages.

The amount of polyether required to produce the desired antisoil redeposition result is from about 0.25 to about 5 percent by weight, based on the weight of the polyester. Fibers containing less than about 0.25 percent of the polyether discolor under normal laundering conditions nearly as badly as the unmodified fibers. Increasing the concentration of the polyether above about 5 percent offers no additional advantages.

The following examples are intended to illustrate my invention so that it may be easily understood but not to limit it in any way.

Example 1

45 grams of poly(ethylene glycol) distearate prepared from a polyethylene glycol (Carbowax 4,000) and stearic acid, were dissolved in 350 milliliters of toluene. The solution was added with stirring to 2,200 grams of pulverized polyester, poly(1,4-cyclohexylenedimethylene terephthalate) of inherent viscosity of 0.82. The mixture was then dried at 60° C. and 0.5 mm. of mercury for three hours and melt spun into fibers which contained 2 percent by weight of the polyether additive. After drafting and heat setting, the yarn was knitted into a sock. A sample of the sock was subjected to a washing in the presence of heavily soiled cotton. After three washings, the sample was not discolored. A control sample of poly(1,4-cyclohexylenedimethylene terephthalate) with no poly(ethylene glycol) distearate was dark gray in color after similar washings.

Example 2

The procedure of Example 1 was followed with the exception that poly(1,4-cyclohexylenedimethylene terephthalate) was replaced with poly(ethylene terephthalate). Equally good results were obtained after three washings when poly(ethylene glycol) distearate was added to the polyester. The sample containing no poly(ethylene glycol) distearate was discolored after three washings.

Example 3

Poly(ethylene glycol) distearate prepared as in Example 1 was injected into a molten stream of poly(1,4-cyclohexylenedimethylene terephthalate) and agitated therewith while molten. The feed rates were adjusted so that 2 percent by weight of the poly(ethylene glycol) distearate was present in the final polymer which had an inherent viscosity of 0.79. The final polymer was melt spun into continuous filament which was drafted, heat set, and converted into 3-inch staple. The staple was then converted into staple yarn and woven into fabric on a loom. The material so prepared did not discolor after repeated washings in a home washer and dryer in the presence of heavily soiled cotton. Example 1 indicates that untreated poly(1,4-cyclohexylenedimethylene terephthalate) becomes discolored under similar laundering conditions.

Example 4

A polyester [poly(1,4-cyclohexylenedimethylene terephthalate)] was pulverized and divided into three equal portions. One percent by weight of poly(ethylene glycol) diacetate was added to another portion of the polyester. Both of the poly(ethylene glycol) esters were prepared from a poly(ethylene glycol) with a molecular weight of 4,000 and the corresponding acid. The two samples of polyester containing the additives as well as the untreated sample were spun into fibers. The fibers were drafted, heat set, and woven into socks as described in Example 1. The socks were all laundered in the presence of heavily

soiled cotton. The results of the laundering test are shown in Table I.

TABLE I

Polymer Composition	Additive	Laundering Test ¹
Poly(1,4-cyclohexylenedimethylene terephthalate).	None.....	5-4+
Do.....	1% poly(ethylene glycol) distearate.	2-1
Do.....	1% poly(ethylene glycol) diacetate.	3-4

¹ The test consisted of 8 separate laundering cycles. Soiled cotton fabrics were added at the beginning of each cycle. The ratings were based on a 1 to 5 scale with the 1 rating being best appearance. The first number in the column represents the rating received after the eighth cycle.

The results of these tests indicate that fibers containing poly(ethylene glycol) diacetate are somewhat superior to fibers without an additive, but not nearly as good as fibers containing poly(ethylene glycol) distearate.

Example 5

A polyester [poly(1,4-cyclohexylenedimethylene terephthalate)] was pulverized and divided into four equal portions and mixed with the amount and composition of the additives shown in Table II.

The four samples were spun into fibers as in Example 1 which were drafted, heat set, and woven into socks as described in Example 1. The socks were laundered in the presence of heavily soiled cotton. The results of the laundering test are shown in Table II.

TABLE II

Polymer Composition	Additive	Laundering Test ¹
Poly(1,4-cyclohexylenedimethylene terephthalate) plus 0.25% TiO ₂ .	None.....	5
Do.....	1% poly(ethylene glycol) distearate.	1
Do.....	1% polypropylene glycol.	4
Do.....	2% polypropylene glycol.	3
Do.....	2% poly(ethylene glycol).	3

¹ The test consisted of washing the socks in the presence of heavily soiled cotton. The ratings were based on a 1 to 5 scale with 1 having the same white appearance as the starting fabric and 5 representing a gray fabric.

The results indicate that the fibers containing poly(ethylene glycol) distearate were much more resistant to adsorption of soil during laundering than were other fibers.

Example 6

A polyester [poly(ethylene terephthalate)] was pulverized and divided into 6 equal portions. One portion was used as a control. An additive was added to each of the other portions. The 6 samples were converted into socks as in Example 1.

The antisoiling additives and the laundering characteristics of the socks are shown in Table III. The data indicates that poly(ethylene glycol) esters of acids containing 10 or more carbons are superior to esters of acids containing less than 10 carbons.

TABLE III

Additive	Laundering Test
None.....	5
1% poly(ethylene glycol) diacetate.....	3
1% poly(ethylene glycol) dicaproate.....	3
1% poly(ethylene glycol) dicaprate.....	2
1% poly(ethylene glycol) dilaurate.....	1
1% poly(ethylene glycol) distearate.....	1
1% poly(ethylene glycol) dibehenate.....	1

Example 7

A polyester [poly(1,4 - cyclohexylenedimethylene terephthalate)] was pulverized and divided into eight equal parts. An additive (1% based on the weight of the polyester) was added to each part, and the polyesters were converted into socks as in Example 1.

The antisoiling additives and the laundering characteristics of the socks are shown in Table IV.

The data indicates that the molecular weight of the poly(ethylene glycol) should be at least 2,000.

TABLE IV

Additive	M.W. of PEG	Laundering Test
1% poly(ethylene glycol) diacetate.....	600	5
Do.....	1,000	4
Do.....	2,000	3
Do.....	4,000	3
1% poly(ethylene glycol) dilaurate.....	600	3
Do.....	1,000	2
Do.....	2,000	1
Do.....	4,000	1

Example 8

A polyester [poly(1,4-cyclohexylenedimethylene terephthalate)] was pulverized and divided into four equal parts. Poly(ethylene glycol) distearate was added to each portion, and the polyesters were converted into socks as in Example 1.

The amounts of poly(ethylene glycol) distearate added to each portion and the laundering characteristics of the socks are shown in Table V.

The results indicate that at least about 0.25% of the additive is needed to impart the desired laundering characteristics.

TABLE V

Additive	Laundering Test
0.15% poly(ethylene glycol) dilaurate.....	3
0.25% poly(ethylene glycol) dilaurate.....	2
0.5% poly(ethylene glycol) dilaurate.....	2
1.0% poly(ethylene glycol) dilaurate.....	1

As indicated above, the particular polyester composition, the method of preparing the fiber-forming polyester polymer, and the method of spinning the polymer into fibers are not limitations on the present invention. Also, as explained in detail in the above examples, there are several useful ways in which the polyether additives of the present invention may be incorporated on and into the polyester fiber-forming polymer compositions. However, if further information is desired concerning certain of the polyesters we prefer to use the melt spinning thereof, such may be had by reference to Kibler et al. U.S. Patent 2,901,466. Particular reference is made to Examples 53 and 54 of this patent concerning useful methods of melt spinning certain preferred polyester compositions.

As can be seen from the foregoing description, this invention provides an especially advantageously new and useful polyester fiber having dispersed therein from about

0.25 to about 5 percent by weight, based on the weight of said polyester fiber, of a fatty acid ester of a poly(ethylene glycol), said fatty acid having 10 to 22 carbon atoms and said poly(ethylene glycol) having a molecular weight of at least 2,000.

As can further be seen from the foregoing description, this invention provides an especially advantageous new and useful process for treating polyesters, said process comprising dispersing therein from about 0.25 to about 5 percent by weight, based on the weight of said polyester, of a fatty acid ester of a poly(ethylene glycol), said fatty acid having 10 to 22 carbon atoms and said poly(ethylene glycol) having a molecular weight of at least 2,000.

As can still further be seen from the foregoing description, this invention provides an especially advantageous new and useful process for preparing a soil resistant polyester fiber, said process comprising (a) mixing a polyester powder with from about 0.25 to about 5 percent by weight, based on the weight of said polyester, of a fatty acid ester of a poly(ethylene glycol), said fatty acid having 10 to 22 carbon atoms and said poly(ethylene glycol) having a molecular weight of at least 2,000, and (b) melt spinning said soil resistant polyester fiber therefrom.

This invention has been described in detail with particular reference to preferred embodiments thereof, but it will be understood that variations and modifications can be effected within the spirit and scope of the invention as described hereinabove and as defined in the appended claims.

I claim:

1. A process for preparing a soil resistant polyester fiber, said process comprising (a) mixing a polyester powder with from about 0.25 to about 5 percent by weight, based on the weight of said polyester, of a fatty acid ester of a poly(ethylene glycol), said fatty acid having 10 to 22 carbon atoms, said poly(ethylene glycol) having a molecular weight of at least 2,000 and said polyester being prepared from at least one diol and at least one dibasic acid, (b) melt spinning said soil resistant polyester fiber therefrom.

2. A process for preparing a soil resistant polyester fiber as defined in claim 1 wherein said fatty acid ester is poly(ethylene glycol) distearate.

3. A process for preparing a soil resistant polyester fiber as defined in claim 1 wherein said polyester is selected from the group consisting of poly(ethylene terephthalate) and poly(1,4-cyclohexylenedimethylene terephthalate).

4. A soil resistant polyester fiber prepared by a process as defined in claim 1.

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