Sept. 3, 1968 G. FARROW 3,400,187

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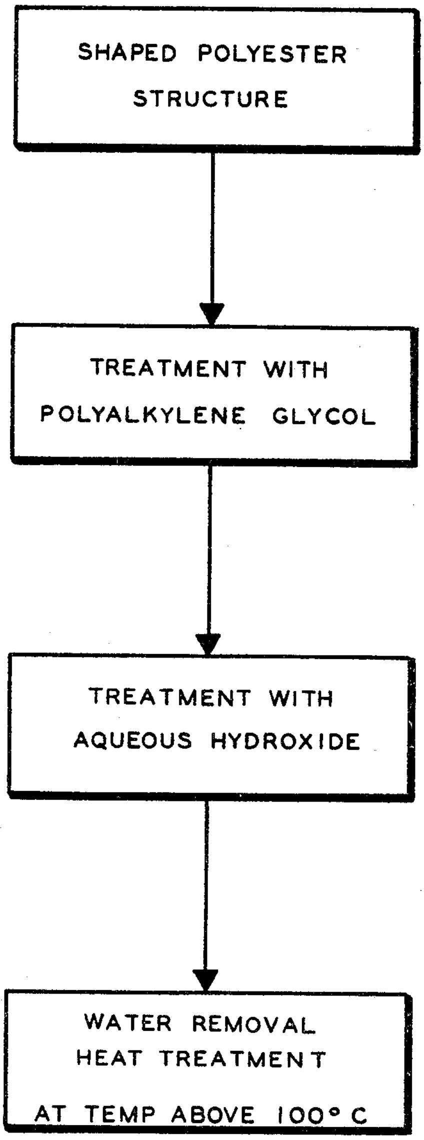
Filed Feb. 25, 1964



INVENTOR
GERALD FARROW

BY

ATTORNEY

3,400,187
Patented Sept. 3, 1968

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METHOD OF TREATING POLYESTER STRUCTURES WITH POLYALKYLENE GLYCOL AND A METAL HYDROXIDE

Gerald Farrow, Kings Mountain, N.C., assignor to Fiber Industries, Inc., a corporation of Delaware

Filed Feb. 25, 1964, Ser. No. 347,107
9 Claims. (Cl. 264—137)

ABSTRACT OF THE DISCLOSURE

A process for improving the surface characteristics of shaped polyester structures comprising the steps of applying to said polyester structure a polyalkylene glycol having a molecular weight in excess of about 200, applying an aqueous metal hydroxide solution and then heating the thus treated polyester structure at temperatures of at least about 100° C. for a time sufficient to remove substantially all of the water.

This invention relates to a process for improving surface characteristics of structures prepared from high molecular weight synthetic linear polyesters. More particularly, the invention is directed to a process for preparing shaped polyester structures having a permanently modified surface to provide property improvements including, among others, desirable hydrophilic characteristics, improved antistatic properties, bonding by adhesives and sizes are significantly increased and improved cleansing properties to remove oils and grease.

The use of polymeric polyester in film production, in production of fibers and filaments, and the like, utilized in the textile field for fabric production as well as industrial uses, have found wide commercial acceptance because of some of their outstanding properties. Although these polyester products produced are considered satisfactory, there are major disadvantages associated with these products arising from the non-polar and hydrophobic nature of their surfaces. These disadvantages include, for example, the great ease with which static electricity is generated and reacted on the shaped articles arising from the poor surface conductivity in view of the difficulty of bonding antistatic agents onto the surfaces under conditions which would make the coatings durable to water washing and organic solvent action; the difficulties of bonding such shaped articles as filaments and fibers together by adhesives and textile sizes and to other substrates; the difficulties of causing printing inks to set and adhere to the shaped articles; and finally the difficulties of causing water to displace soils both by oily and greasy substrates from the surfaces of the shaped articles during washing and detergency.

Numerous methods have been proposed for treating polyester structures such as films, filaments, fibers and the like, to overcome the above-described disadvantages. Among the known processes, the commonly used method for treatment of surfaces of polyester structures utilize spin finishes, antistatic finishes and the like, to provide temporary improvements of these problems; however, these methods do not attach the treating compounds permanently to the surface and are readily removed on contact with suitable solvents. For example, washing and dry-cleaning processes easily remove many of these treating compounds which are merely temporarily adhered to or dissolved in the surface of the article. In order to prevent removal of improved surface characteristics of polyester structures by solvents or water, a form of a chemical graft of treating compounds to the surface of the structure appears essential to provide the permanent desirable surface characteristics. Some proposals have been suggested to provide these improvements, but none has provided a commercially feasible process.

The over-all object of this invention is to provide a process for preparing shaped polyester structures having improved permanent surface characteristics. A further object is to provide a commercially feasible process for preparing polyester structures in which the surface of said structure is modified to provide permanently improved antistatic properties, permanently improved cleansing properties and permanently improved bonding characteristics, and the like. Numerous other objects will become apparent to those skilled in the art from a consideration of the disclosure.

Accordingly, the process of this invention relates to the treatment of shaped polyester structures by initially applying polyalkylene glycols to the surface of the structures. After the polyalkylene glycol has been applied, an aqueous hydroxide solution of an alkaline earth metal or an alkaline metal is additionally applied to the shaped structures followed by heating the combination of the polyalkylene glycol and aqueous hydroxide solution at temperatures in the range from about 100° C. to below the softening point of the polyester structure for a period of time sufficient to remove substantially all of the available water. The resulting product of this process is one which has a permanent surface modification, chemically adhered or grafted to the shaped polyester structure. It is of significance that this simple and unique process can provide polyester shaped structures having permanently improved hydrophilic properties, improved antistatic properties, as well as improved cleaning properties after repeated washings in water and/or detergents or dry-cleaning solutions. It should be readily apparent that the process of this invention is economically feasible to prepare polyester fibers or filaments, and the like, having the highly desirable, permanent properties described above for use especially in the textile apparel field. In addition to these significant improvements, bonding properties of the resulting products in the use of adhesives or sizes are significantly improved which are of considerable interest in industrial uses for the polyester products.

A better understanding of the invention may be had from the drawing which is a flow sheet of the process of this invention. As can be noted from the flow sheet, the shaped polyester structure is subjected to a polyalkylene glycol treatment followed by an aqueous hydroxide solution treatment, followed by heating at temperatures in excess of about 100° C.

The polyester shaped structures used in the process of this invention can include a variety of synthetic, highly linear polymers which are capable of being spun or extruded and then stretched for orientation into a synthetic fiber, filament, yarns, thread, film and the like. Examples of the compositions of the shaped structures can include, among others, linear polyesters of aromatic dicarboxylic acids and dihydric compounds such as polyethylene terephthalate, and ethylene glycol, copolymers derived from terephthalic acid and bis-1,4(hydroxymethyl) cyclohexane; modifications of such polyesters including an additional monomer component such as sebacic acid, isophthalic acid, organic mono- and dicarboxylic compounds containing at least one metallic salt sulfonate radical. In addition to the above-described polyester shaped structures, additional structures can include fabric blends of polyester with wool, cotton, among other natural fibers, as well as other suitable synthetic fibers.

The polyalkylene glycols which are utilized herein are those having average molecular weights in excess of about 200; and the average molecular weights can range from about 200 to about 17,000, preferably in the range from about 200 to about 6,000. It has been observed that in utilizing polyalkylene glycols having average molecular weights below about 200, no specific reaction occurs utilizing this process. The amount of polyalkylene glycol applied to the shaped structure can range from about 0.05 percent to about 10 percent or higher by weight of the shaped structure. The type of polyalkylene glycol which can be utilized includes polyethylene glycol, polypropylene glycol and mixtures thereof; or copolymers of said polyethylene or polypropylene glycols in which are incorporated, among others, such as additional alkyl alcohols, aryl alcohols, alkyl aryl alcohols, phenols, amides, imides, amines, polyfunctional alcohols, and the like. The preferred polyalkylene glycol is one which is preferably soluble in water. Although the polyalkylene glycol can be applied to the polyester shaped structure in concentrated form in the form of a liquid or solid, the use of liquid and especially an aqueous solution containing at least about 2 percent by weight of the polyalkylene glycol will permit a more uniform application on the surface of the treating structures.

The polyalkylene glycols can be applied on the surface of the shaped polyester structure at any time in the processing procedure, but it is essential that the polyalkylene glycol application be conducted prior to the aqueous alkaline hydroxide or alkaline earth metal hydroxide application. The application of polyalkylene glycols can be made prior to or after the drawing or stretching stage. It is preferred, however, to apply the polyalkylene glycol prior to the drawing or stretching operation since the presence of these polyalkylene glycols will provide added lubricating effects in the drawing or stretching operation. The polyalkylene glycols can be applied on the surface of the shaped structures at temperatures ranging from about room temperature, or below, to below the temperatures wherein the shaped structure may be detrimentally affected. As is known, the stretching of undrawn structures such as yarns or films is generally carried out at temperatures in excess of the second order transition temperature to about 50° C. above the second order transition temperature of the structure. The amount of stretching is controlled to obtain a draw ratio of from about 2:1 to about 8:1 to obtain the desired physical properties of the finished structure. The method of applying the polyalkylene glycol is not necessarily critical and can consist of padding, spraying, utilization of an applicator roll, or the like. The amount of polyalkylene glycol applied to the surface of the shaped polyester structure is dependent on many factors, for example, manner of application, type of surface to which it is applied, among other methods. The amount of polyalkylene glycol applied to the shaped polyester structure can range from about 0.1 to about 2.0 percent by weight of the shaped structure. Preferably in the range from about 0.25 to about 0.85 percent in the dried form.

After the polyalkylene glycols have been applied, the aqueous hydroxide solution of alkali metals, such as sodum, potassium, lithium and the like, and alkaline earth metals such as barium, calcium, and the like, are applied to the surface of the polyester shaped structures. As in the application of the polyalkylene glycols, the hydroxide solutions can be applied at any stage of the process as long as it follows the polyalkylene glycol application. The preferred temperatures of the hydroxide solutions for application are room temperature, i.e. about 23° C. or below, and for the undrawn or unstretched structures the temperatures should not exceed the second order transition temperature of the polyester structures. After the stretching or drawing step, temperatures to about 100° C. can be utilized, if desired. The concentration of aqueous alkali hydroxides or alkaline earth hydroxides used in the application can range from about 0.01 percent to about 25 percent by weight, or higher, preferably in the range from about 0.1 weight percent to about 5 weight percent; i.e. hydroxide to water. The method of applying the hydroxide solutions is not necessarily critical and can include padding, spraying, utilization of an applicator roll, and the like.

After the combination of polyalkylene glycol and hydroxide solutions have been applied to the surface of the shaped polyester structures, the resulting product is heated in the range from about 100° C. to below the softening point of the structure for a sufficient period of time to remove substantially all of the available water. The time of heating can range from fractions of a second up to as high as 5 minutes, if desired. It is highly desirable that after the hydroxide solution is applied, the heating of the product be conducted at the earliest possible moment to avoid the possibility of forming intermediates, such as carbonates, which may prevent the desired chemical reaction of the metal hydroxide with the polyalkylene glycol which, in turn, reacts with the polyester shaped stuructures. It has been observed that if prior to the heating, extended delays exist after the application of the hydroxide solution, a satisfactory modified surface of the product may not be obtained. Additionally, the mixture of alkali or alkaline earth hydroxide in the polyalkylene glycols for application to the surface of the polyester structure does not provide a satisfactory modified surface for purposes of this invention in view of the production of alkali or alkaline earth carbonates. The method of heating the resulting product is not necessarily critical and any convenient method such as a hot plate, hot roll, heating tube, radiant heating, and the like, can be used.

The resulting product of the process of this invention is a shaped polyester structure having a modified surface which is chemically bonded to the shaped structure to provide a new surface film which is durable to many washings and solvent treatments and has a high affinity for water and polar substances compared with the original surface of the shaped article. Furthermore, the tendency of the resulting shaped polyester product to generate static electricity is greatly reduced; the bonding of the shaped product by adhesive and sizes is considerably increased; the wetting and adhesion of printing inks is improved; and the ease with which water will displace air; oils and grease from the surface of the product is greatly increased.

While not wishing to be bound thereby, it is believed that the alkali metal or alkaline earth metals react on heating with the polyalkylene glycols and, on the removal of water, form the corresponding metal polyalkylene anion which in turn chemically reacts with a polyester structure to form the desired modified surface. In any event, by the process here proposed, a highly desirable permanent modified surface is obtained for the polyester shaped structures, with a minimum loss of the physical properties of the original structure, which overcomes many difficulties long existing in the textile art in the use of polyester products.

The following examples will serve to illustrate the invention without limiting the same:

EXAMPLE 1

Polyethylene terephthalate semi-dull drawn yarn (70 denier–15 filaments) is passed over an applicator wheel which contains polyethylene glycol having an average molecular weight of about 400. The temperature of the polyethylene glycol is maintained at room temperature during the application. The amount of polyethylene glycol in the yarn is found to be 2.8 weight percent of the total yarn. This yarn is then passed over an applicator wheel containing aqueous sodium hydroxide solution having a concentration of sodium hydroxide of 3.4 weight percent. The temperature of the sodium hydroxide solution is maintained at room temperature during the application. After the sodium hydroxide application, the yarn is passed over a 10 inch hot plate maintained at 190° C.

at a speed of 500 feet per minute and wound onto a bobbin.

In a similar manner as described above, polyethylene terephthalate yarn is treated with polyethylene glycol but the sodium hydroxide treatment is eliminated, followed by the hot plate treatment. Additionally, polyethylene terephthalate yarn is only treated with the sodium hydroxide solution eliminating the application of the polyethylene glycol followed by the hot plate treatment.

Samples of each of the yarn are then knitted into individual hoselegs. Part of each hoseleg is washed for 20 minutes in running hot water (65° C.) and another part of each hoseleg is washed utilizing agitation for 30 minutes in boiling water containing a 0.5 weight percent solution of a detergent, made and sold by Proctor & Gamble Company, Cincinnati, Ohio, under the trademark Tide. All of the hoselegs are rinsed and dried utilizing the same procedure. All of the hoselegs are folded in the same manner and droplets of water, from a hypodermic syringe, are placed carefully on each of the individual surfaces. The hoseleg samples which are treated with the polyethylene glycol followed by the sodium hydroxide treatment adsorb water rapidly and are hydrophilic. The remaining hoseleg samples do not possess the hydrophilic properties since the water remains on the surfaces until it evaporates.

EXAMPLES 2–6

Polyethylene terephthalate semi-dull drawn yarn (70 denier–36 filaments) are treated utilizing the following procedures:

Example 2—A portion of untreated yarn is passed over a 10 inch hot plate maintained at temperatures of 190° C. to 200° C.

Example 3—The yarn is passed over an applicator wheel containing an aqueous solution of 50 weight percent polyethylene glycol having an average molecular weight of 1000 at room temperature and passing the yarn over a 10 inch hot plate maintained at a temperature of 190° C. to 200° C.

Example 4—The yarn is passed over an applicator wheel containing an aqueous solution of 25 weight percent polyethylene glycol having an average molecular weight of 1000 at room temperature and passing the yarn over a 10 inch hot plate maintained at a temperature of 190° C. to 200° C.

Example 5—The yarn treatment of Example 3 except that prior to the hot plate treatment, the yarn is passed over an applicator wheel containing an aqueous solution of 5 weight percent sodium hydroxide maintained at room temperature.

Example 6—The yarn treatment of Example 4 except that prior to the hot plate treatment, the yarn is passed over an applicator wheel containing an aqueous solution of 5 weight percent sodium hydroxide maintained at room temperature.

Each of the yarns of Examples 2 through 6 are knitted into individual hoselegs. A portion of each hoseleg sample is washed in hot water and the remaining portion washed five separate times for 30 minutes in a boiling water solution containing 0.5 percent detergent (Tide). The results relating to the hydrophilic and antistatic properties of the samples are described in Table I below:

TABLE I

	Time in seconds for adsorption of water droplets		Resistance, megohms of hoseleg 65% relative humidity*	
	Washed 20 minutes hot water, 65° C.	5 washes in "Tide," 100° C.	Washed 20 minutes hot water, 65° C.	5 washes in "Tide," 100° C.
Example 2 (No treatment)	∞	∞	$>50\times10^6$	$>50\times10^6$
Example 3 (Polyalkylene glycol treatment only)	∞	∞	$>50\times10^6$	$>50\times10^6$
Example 4 (Polyalkylene glycol treatment only)	∞	∞	$>50\times10^6$	$>50\times10^6$
Example 5 (Polyalkylene glycol & sodium hydroxide treatment).	8	8	$0.3\text{–}1.2\times10^5$	$0.3\text{–}2\times10^6$
Example 6 (Polyalkylene glycol & sodium hydroxide treatment).	10	10	10.5×10^4	$0.3\text{–}2\times10^6$

*Measuring resistance using a 50 million megohm electrical resistance tester manufactured by Custom Scientific Instruments, Inc., Kearny, N.J. (See reference: Hayck, M. and Chromey, F. C. American Dyestuff Reporter, April, 1951. A.A.T.C.C. Standard Test Method 76–1959).

It should be noted from the above results that significant improvements in antistatic properties as well as hydrophilic properties are demonstrated in Examples 5 and 6 utilizing the process of this invention.

To the individual yarn products obtained in Examples 3 through 6 described above, an aqueous solution of polyacrylic acid is applied to the surfaces of each of the yarn products. The resulting products are dried in an oven at 110° C. On the removal from the oven, coatings of polyacrylic acid have formed but have completely detached themselves from the yarn products of Examples 3 and 4 wherein the product is only treated with a polyalkylene glycol; whereas, the coating of the same material adheres firmly and uniformly to the yarn products of Examples 5 and 6 produced by the process of this invention. This result demonstrates the improvements of the adhesive effects of the products produced by the process of this invention.

A portion of the yarn products of Examples 3 and 5 are soaked in castor oil and then immersed in warm water containing a trace of detergent. In the case of the product of Example 5 which is produced by the process of this invention, the oil separates into small globules on the yarn product and because of the large contact angle in the oil, it flows away into the washing liquor leaving the surface of the yarn quite free of oil. On the other hand, in the yarn product of Example 3 which is prepared utilizing the polyalkylene glycol only, the oil droplets do not separate but remain as a film on the yarn surface. This oil film is more difficult to remove and requires vigorous abrasion under the wash liquor. This comparison demonstrates how the removal of oils and greases is improved in the product produced by the process of this invention.

Similar improvements as demonstrated in Examples 5 and 6 are obtained for polyethylene terephthalate films.

EXAMPLE 7

An aqueous solution of polyethylene glycol having an average molecular weight of about 4000 and a concentration of 15 weight percent of said polyethylene glycol is applied at room temperature on a polyethylene terephthalate spun yarn of 237 denier. The yarn is passed over a heated drawing roll (80° C.) and stretched approximately 3.3 times its original length. The dawn or stretched yarn is passed over an applicator roll containing an aqueous solution of sodium hydroxide having a concentration of 5 weight percent of sodium hydroxide at room temperature. The amount of sodium hydroxide pick up is found to be of the order of 0.5 weight percent of the yarn. The yarn is then contacted with a 10 inch hot plate maintained at 200° C. From the hot plate the yarn is wound onto a bobbin at a rate of 500 feet per minute. Hoseleg samples are knitted from the yarn product. The hoseleg samples are found to be hydrophilic even after 5 repeated washings in a boiling solution containing 0.5 percent detergent

(Tide) or pure soap. The resistance measurements at 65 percent relative humidity after washing range from 3×10^4 to 3×10^6 meghoms.

For comparison purposes, the above procedure was duplicated except for the sodium hydroxide treatment. The resulting product was completely hydrophobic and provided increased static electricity whereby infinite resistance values, i.e. $>50\times10^6$ megohms were measured.

EXAMPLE 8

An aqueous solution of polyethylene glycol having an average molecular weight of about 4000 and a concentration of 10 weight percent of said polyethylene glycol is applied at room temperature on a polyethylene terephthalate spun yarn of 237 denier. The yarn is passed over a heated drawing roll (80° C.) and stretched approximately 3.3 times its original length. The drawn or stretched yarn is passed over an applicator roll containing an aqueous solution of sodium hydroxide having a concentration of 5 weight percent of sodium hydroxide at room temperature. The amount of sodium hydroxide pick up is found to be to be of the order of 0.5 weight percent of the yarn. The yarn is then contacted with a 10 inch hot plate maintained at 200° C. From the hot plate the yarn is wound onto a bobbin at a rate of 500 feet per minute. Hoseleg samples are knitted from the yarn product. The hoseleg samples are found to be hydrophilic even after 5 repeated washings in a boiling solution containing 0.5 percent detergent (Tide) or pure soap. The resistance measurements of 65 percent relative humidity after washing range from 3×10^4 to 3×10^6 meghoms.

For comparison purposes, the above procedure was duplicated except for the sodium hydroxide treatment. The resulting product was completely hydrophobic and provided increased static electricity whereby infinite resistance values, i.e. $>50\times10^6$ meghoms were measured.

In a similar manner as described above, an aqueous calcium hydroxide solution can be substituted for the sodium hydroxide solution in the process of this invention to obtain the desired product.

EXAMPLE 9

An aqueous solution of polyethylene glycol having an average molecular weight of about 4000 and a concentration of 10 weight percent of said polyethylene glycol is applied at room temperature on a polyethylene terephthalate spun yarn of 237 denier (36 filaments). Two sets of finished levels are applied to the above-spun yarn. In one instance, the finished level of the polyethylene glycol solution applied to the above yarn is 0.5 to 0.7 weight percent of said yarn; and in the other instance, the finished level of 0.8 to 1.1 weight percent is applied. After the polyethylene glycol is applied, the yarns are passed over an applicator roll containing an aqueous solution of sodium hydroxide having a concentration of 5 weight percent of sodium hydroxide at room temperature. The respective yarns are then passed over a heated roll at 80° C. and stretched approximately 3.3 times their original length. After the stretching operation, the yarns are then contacted with a 10 inch hot plate maintained at 200° C. From the hot plate, the yarns are wound onto bobbins at rates of 500 feet per minute. Hoseleg samples are knitted from each of the yarn products. The hoseleg samples made from the yarn products are found to be hydrophilic even after 5 repeated washings in a boiling solvent containing 0.5 percent detergent (Tide) or pure soap. The resistance measurements at 65 percent relative humidity after washing indicate that these yarn products have significantly improved antistatic properties over the non-treated yarns.

In a similar manner as described above, an aqueous solution of a mixture of polyethylene glycol having an average molecular weight of about 4000 plus polyethoxylated glyceride (Atlas G 1288) (soluble in water) in a weight ratio of 6:4 i.e. polyalkylene glycol to polyethoxylated glyceride is prepared to provide a concentration of

10 weight percent of the glycol glyceride solution. This aqueous mixture is applied to two separate yarn products as described above, followed by the sodium hydroxide treatment, drawing or stretching operation plus the heating treatment. The resulting properties of yarn products produced in this manner were similar to those of the yarn products of Example 9.

It is understood that the foregoing description is merely illustrative of preferred embodiments of the invention of which many variations may be made by those skilled in the art within the scope of the following claims without departing from the spirit thereof.

What is claimed is:

1. A process for preparing shaped polyester structures which have permanent modified surface characteristics comprising the steps of the applying polyalkylene glycol having an average molecular weight in excess of about 200 to the surface of said structure, additionally applying an aqueous metal hydroxide solution wherein the metals are selected from the group consisting of an alkali and an alkaline earth metal, and heating the resulting structure at temperatures in the range from about 100° C. to below the softening point of said structure for a period of time sufficient to remove substantially all of the available water.

2. The process of claim **1** wherein the polyalkylene glycol used has an average molecular weight in the range from about 200 to about 17,000.

3. A process for preparing shaped polyester structures which have permanent modified surface characteristics comprising the steps of applying polyalkylene glycol having an average molecular weight in the range from about 200 to about 6000 to the suface of said structure, additionally applying an aqueous sodium hydroxide solution, and heating the resulting structure at temperatures in the range from about 100° C. to below the softening point of said structure for a period of time sufficient to remove substantially all of the available water.

4. A process for preparing polyester yarn which has permanently modified surface characteristics comprising the steps of applying polyalkylene glycol having an average molecular weight in excess of 200 to the surface of said spun polyester yarn; stretching said treated polyester yarn at least from about 2 to about 8 times its original length at temperatures in the range from about the second order transition temperature of said polyester yarn to about 50° C. above said second order transition temperature; applying to the surface of said stretched yarn, aqueous metal hydroxide solution wherein the metal is selected from the group consisting of an alkali and an alkaline earth metal; and heating the resulting polyester yarn at temperatures in the range from about 100° C. to below the softening point of said polyester yarn for a period of time sufficient to remove substantially all of the available water.

5. The process of claim **4** wherein the polyalkylene glycol used has a molecular weight in the range from about 200 to about 17,000.

6. A process for preparing polyester yarn which has permanently modified surface characteristics comprising the steps of applying polyethylene glycol having an average molecular weight in the range from about 200 to about 6000 to the surface of said spun polyester yarn; stretching said treated polyester yarn at least from about 2 to about 8 times its original length at temperatures in the range from about the second order transistion temperature of said polyester yarn to about 50° C. above said second order transition temperature; applying to the surface of said stretched yarn, aqueous sodium hydroxide solution; and heating the resulting polyester yarn at temperatures in the range from about 100° C. to below the softening point of said polyester yarn for a period of time sufficient to remove substantially all of the available water.

7. A process for preparing polyester yarn which has permanently modified surface characteristics comprising the steps of applying polyalkylene glycol having an average molecular weight in excess of about 200 to the surface of said spun polyester yarn; additionally applying to said surface aqueous metal hydroxide solution wherein the metal is selected from the group consisting of an alkali and an alkaline earth metal; stretching said treated polyester yarn at least from about 2 to about 8 times its original length at temperatures in the range from about the second order transition temperature of said polyester yarn to about 50° C. above said second order transition temperature; and heating the resulting polyester yarn at temperatures in the range from about 100° C. to below the softening point of said polyester yarn for a period of time sufficient to remove substantially all of the available water.

8. The process of claim **7** wherein the polyalkylene glycol used as a molecular weight in the range from about 200 to about 17,000.

9. A process for preparing polyester yarn which has permanently modified surface characteristics comprising the steps of applying polyethylene glycol having an average molecular weight in the range from about 200 to about 6000 to the surface of said spun polyester yarn; additionally applying to said surface aqueous sodium hydroxide solution; stretching said treated polyester yarn at least from about 2 to about 8 times its original length at temperatures in the range from about the second order transition temperature of said polyester yarn to about 50° C. above said second order transition temperature; and heating the resulting polyester yarn at temperatures in the range from about 100° C. to below the softening point of said polyester yarn for a period of time to remove substantially all of the available water.

References Cited

UNITED STATES PATENTS

2,955,954	10/1960	Collins	117—47
3,190,718	6/1965	Schoeneberg et al.	8—130.1
2,938,811	5/1960	Hermes.	
2,998,296	8/1961	Hennemann.	
3,110,617	11/1963	Scott.	
3,135,577	6/1964	Watson.	
3,251,913	5/1966	Richards et al.	

FOREIGN PATENTS

758,745	10/1956	Great Britain.

JAMES A. SEIDLECK, *Primary Examiner.*

A. KOECKERT, *Assistant Examiner.*