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METHOD FOR PROCESSING CELLULOSE ESTER YARNS

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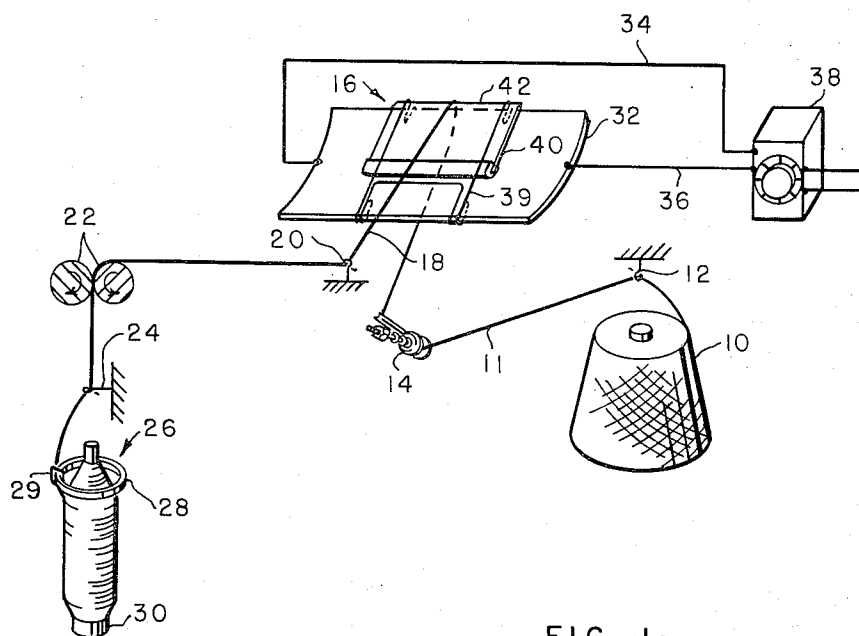


FIG. -I-

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**METHOD FOR PROCESSING CELLULOSE
ESTER YARNS**

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12 Claims. (Cl. 28—72)

This invention relates to improved methods for elasticizing cellulose ester yarns and more particularly the invention relates to improved methods of the type wherein the yarn is passed through a linear path having a sharply angular portion to thereby deform the yarn.

It has been known for a number of years that passing a cold thermoplastic yarn in its normal state over a deforming member will cause the yarn to loop and curl to some extent (see British Patent 558,297) but such a process does not produce a product which can be satisfactorily employed as an elasticized yarn. A primary reason why this is true is that the tendency to loop or curl is lost by placing the yarn in hot or even cold water and fabrics or clothing made therefrom soon lose their elastic nature. In U.S. application, Serial No. 274,358, filed March 1, 1952, it is disclosed that, under the proper conditions, passing a thermoplastic yarn at an elevated temperature through a path having a sharply angular portion will result in yarns having, in most instances, a high degree of elasticity of a very permanent nature. For example, nylon yarns processed in this manner develop a very strong tendency to coil and loop which tendency normally persists for years and is not lost by subjecting the yarns to either hot or cold water. While, however, the method of U.S. patent application, Serial No. 274,358, gives a high measure of elasticity of a permanent nature with most yarns, when the method is employed with the cellulose ester yarns, the tendency to loop and coil is not so permanent as might be desired in many instances.

It is, therefore, a primary object of this invention to provide an improved non-torque method for producing cellulose ester yarns or fibers having a pronounced tendency to loop and curl with a greater degree of permanency than heretofore obtainable.

The above, as well as other objects of the invention, are accomplished by a process which comprises treating a cellulose ester yarn with a reagent which acts to increase the elongation to break of the yarn without appreciably decreasing the strength of the yarn. Suitable reagents which can be employed for this purpose include the non-oxidizing mineral acids, as illustrated by sulfuric and hydrochloric acid, salts of such acids with polyvalent metals, or mixtures of said salts and said acids. Salts with metals of groups 1B, 2B, 3A and 4A of the periodic table are especially advantageous and suitable salts may be illustrated by zinc chloride, cupric chloride, aluminum chloride, stannic chloride, and cadmium sulfate. The presently preferred reagent is zinc chloride.

The process of this invention is suitable for use with yarns or the like composed of any cellulose ester or mixed ester as illustrated by cellulose acetate, cellulose acetate propionate and cellulose acetate butyrate, but since cellulose acetate yarns are the only members of this family to have received widespread commercial acceptance, the invention will be described with particular reference to such yarns. Cellulose acetate yarns having any desired acetyl content can be employed although the higher the acetyl content of the yarn, the more hydrophobic is the

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yarn and the better are the results obtained when the yarn is processed according to this invention. For best results the acetyl content of the yarn should be at least about 35.8% by weight and preferably about 44.8% by weight. Commercial "cellulose acetate" yarns generally contain about 39% acetyl whereas the acetyl groups in cellulose triacetate constitute 44.8% of its weight.

The denier and filament size of yarns or the like employed in the improved process of this invention may vary within wide limits and, in fact, yarns of almost any total denier or filament size can be suitably employed. By way of illustration, the process can be employed with excellent results with 300 denier 80 filament diacetate or triacetate yarn, 75 denier 20 filament diacetate or triacetate yarn or 1200 denier 160 filament diacetate or triacetate yarn. Under suitable conditions the denier per filament can range from about 0.5 to 20 and the total denier of the yarn can readily be as high as several thousand. In fact, it is expected that the invention will have its greatest utility in the elasticization of a cellulose acetate tow containing thousands of individual filaments.

The acid and/or salt reagent is preferably employed in aqueous solution and solutions having a concentration of from about 0.05 M to 0.4 M can suitably be utilized. The preferred concentration for the aqueous solution is generally from about 0.08 M to 0.15 M depending upon the particular acid or salt employed and other considerations. The reagent solution can be employed at any suitable temperature limited only by the freezing and boiling points of the solution under the particular conditions of use, but for reasons of convenience, the solution is preferably applied to the yarn at room temperature.

The time that the cellulose ester yarn is acted upon by the reagent solution may be varied within wide limits and the yarn may be only momentarily immersed in the reagent solution to gain a slight measure of improvement; however, for best results, the solution should generally be allowed to act upon the yarn for the maximum time possible without severely weakening the yarn or, in other words, without the yarn suffering a loss in tensile strength of more than about 10 to 20%. The exact length of time that the reagent can be allowed to act upon the yarn without severe loss of strength depends upon the concentration of the reagent solution and also upon the temperature of application, but when employing room temperature and a 0.1 M solution of the reagent, best results are generally obtained by treating the yarn for a period of from between about 3 to 5 hours. With increased reagent concentrations and/or with increased temperatures of application, the time of treatment should be correspondingly shortened.

In addition to the salt and/or acid, the reagent solution can contain other materials added for various purposes. For example, the solution may additionally contain a wetting agent to obtain faster penetration of the yarn by the solution or it may contain an organic swelling agent, such as ethylene glycol dimethyl ether, to assist in swelling the cellulose ester yarn. Frequently end results are somewhat improved by adding an organic solvent to assist in removing the antistatic agent from the yarn so that it ribbons to a greater extent in subsequent operations.

The reagent solution may be applied by any suitable procedure and may be applied to a running single end of yarn or may be applied to the yarn while in package form. When employing temperatures and reagent concentrations such that a relatively long period of treatment is required, it is generally advantageous to treat the yarn while in package form. This can be accomplished by merely soaking a loosely wound package of the yarn in a solution of the reagent but for more uniform results, the reagent is preferably pumped through the pack-

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age in a manner similar to that conventionally employed in dyeing or the like.

Following the treatment with the acid and/or salt, the yarn should be thoroughly washed to remove as much of the reagent as is practical, and in instances where the reagent solution comprises sulfuric acid, it is generally advantageous to rinse the yarn in a neutralizing basic solution. Sulfuric acid is extremely difficult to remove from cellulose ester yarns and may cause deterioration in time if not adequately neutralized. The rinsing operation is also preferably performed while the yarn is in package form but may, if desired, be conducted upon a running length of yarn. The yarn is then preferably dried to remove excess moisture before further processing operations are performed.

Reference will now be made to the accompanying drawing which schematically illustrates suitable apparatus for passing the treated yarn under tension through a sharply angular path. The reference numeral 10 indicates a means to supply an end 11 of cellulose ester yarn treated as above described. The yarn end 11 is led from supply package 10 through a guide eye 12 and about a tension regulating device 14 to a blade assembly generally indicated by the reference numeral 16. The tension regulating device 14 serves the dual purpose of removing fluctuations in tension resulting from the removal of the yarn from the supply package 10 and of supplying the yarn end 11 to the blade assembly 16 at a proper tension, while the guide 12 makes possible the removal of the yarn end 11 from the yarn supply package 10 in an over-end manner.

From the blade assembly 16, which will be subsequently described in greater detail, the yarn end 11 is drawn through a portion of the yarn path at 18 having a relatively great radius of curvature and is then passed through a guide 20 to a pair of driven rolls 22. From rolls 22 the yarn passes through a guide 24 to a conventional take-up means 26 here illustrated as comprising a conventional ring 28, traveler 29 and bobbin 30.

The blade assembly, generally indicated by the reference numeral 16, is illustrated as comprising a heater strip 32 bent to a radius of about 4 inches in order to present a slightly curved surface to the yarn. The heater strip 32 can be heated by any suitable means such as by the passage therethrough of an electrical current and is connected by a pair of electrical conductors 34 and 36 to a variable transformer 38 which is supplied with power from any suitable source. Mounted on heater strip 32 by means of a holder 39 is a blade member 40 here illustrated as comprising a common razor blade. The edge 42 of blade 40 extends beyond the heater strip 32 a short distance so that the yarn end 11 passes in contact with the under side of the strip and over edge 42 of the blade 40 in an angular path with the edge 42 positioned at the apex of the angle.

The radius of curvature of the blade edge 42 can vary within reasonably wide limits but is preferably as small as is possible without severing the yarn. The smallest possible radius of curvature of the blade in turn depends upon the size of the filaments in the yarn being processed and upon the texture of the material from which blade is formed. With a blade formed from a finely grained material it is frequently possible for the radius of curvature of the edge to be as small as about 4 to 8 microns when processing yarns composed of filaments of about 2 denier or less but with larger filaments or with a blade composed of a coarser grained material, the radius of curvature of the edge should generally be at least about 8 to 16 microns. The maximum radius of curvature of the blade edge depends primarily upon the size of the yarn filaments being passed thereover and as a general rule the radius of curvature should be no more than about 3 to 8 times the diameter of the yarn filaments.

The angle of approach and angle of departure of the yarn to the blade may also vary within wide limits al-

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though the yarn should undergo a change of direction of at least about 45° and preferably at least about 90° in passing about the edge 42. It is generally advantageous to make the angle of approach relatively large, for example, 30 to 100°, so that the blade is displaced from the heater element and is, therefore, at a lower temperature, and it is also generally advantageous that the angle of departure be as small as the grind of the blade edge will permit so that the yarn contacts the face of the blade in being withdrawn from the edge 42.

The tension in the yarn passing over the blade edge is also an important factor and should generally be as high as is possible without resulting in an excessive number of ends down. The strength of cellulose ester yarns is such that it is seldom possible to pass the yarn about the blade edge under a tension of more than about 0.4 gram per denier and in order to reduce the number of ends down it is frequently necessary that the tension of the yarn passing about the edge of the blade be as low as about 0.2 to 0.3 gram per denier. Operative results can, in some instances, be achieved by employing tensions as low as about 0.05 gram per denier but it is a general rule that the higher the tension, the greater the degree of elasticization obtained and sufficient tension should generally be employed to result in a noticeable elongation of the yarn as it passes about the blade edge.

The linear velocity of the yarn over the blade edge may be varied depending upon a number of factors, but as a general rule is preferably as high as equipment limitations will permit. In some instances it may be necessary to employ a low linear yarn velocity in order for the yarn to be adequately heated if a very narrow heater strip is employed and in other instances a low linear yarn velocity may be required because of the limitations of the take-up device. With suitable apparatus it is possible to pass the yarn about the edge at any linear speed up to about 200 to 500 yards per minute or even higher.

The distance that the yarn is in contact with the heater element should be sufficient to insure that the yarn uniformly reaches a suitable temperature for elasticization, but on the other hand should not be so great that the yarn is tensioned excessively by contact with the heater element. If the linear yarn velocity is below about 50 to 80 yards per minute, adequate heating of the small denier yarns can generally be achieved by passing the yarn in contact with the heater element for about 1 inch, but with yarns above about 100 denier or with linear yarn velocities above about 80 yards per minute, the distance the yarn is in contact with the heater element should be correspondingly greater. A length of contact of from about 3 to 9 inches is generally adequate for all conditions, although in some instances, for example, when processing a continuous filament tow of several thousand denier, it may be advantageous to retain the yarn in contact with the heater element a distance as great as about 20 inches.

The temperature of the yarn as it passes into contact with the blade edge should be at least below the sticking temperature of the yarn and operative results can be achieved with the yarn at temperatures as low as about 200° F. As a general rule, however, the higher the temperature the greater the degree of elasticization obtained and the more permanent is the tendency of the yarn to assume a coiled configuration so that the yarn is preferably heated to as high a temperature as it is possible to achieve without excessive damage to the yarn. The preferred temperature range for cellulose acetate yarns is from about 360 to 440° F.

Following its contact with the blade edge the yarn should be cooled as rapidly as possible. It is a general rule that the temperature of the yarn should be reduced until it is at least about 80° F. below the temperature at which it is passed to the blade edge and preferably the temperature of the yarn is rapidly lowered until it

is at least about 150° below the temperature at which it contacts the blade edge.

The radius of curvature of the portion of the yarn path immediately following the point where the yarn passes about the blade edge should be large as compared to the radius of curvature of the blade edge itself. If one is employing a flat blade and the yarn is passed in sliding contact with one face thereof, the radius of curvature of this section of the yarn path will, of course, be infinite and such is generally preferred although operative results can be achieved when this portion of the yarn path has a radius of curvature equal to no less than about 10 to 50 times the radius of curvature of the blade edge. The length of this portion of the path need not be great and adequate cooling of the yarn can generally be accomplished in $\frac{1}{8}$ of an inch or less although a length of one inch or more is generally preferred.

As with other edge elasticized yarns, the tendency to coil which is imparted by passing the yarn about the blade edge is partially latent in nature and can be further developed by heating the yarns in an untensioned condition. A high temperature is not required and the crimp can generally be fully developed by heating the yarn to a temperature of only about 120 to 140° F. for a few seconds. The heating can be accomplished by passing the yarn into an aqueous bath maintained at an elevated temperature, by means of steam or by means of dry heat. The use of dry heat is generally preferred in the process of this invention since it eliminates the requirement of subsequently drying the yarn and since it generally results in a higher degree of elasticization.

The invention will now be illustrated by the following specific examples:

Example I

One of a plurality of cones of 300 denier 80 filament cellulose triacetate yarn wound upon porous cores is treated with 0.1 M zinc chloride for 15 minutes at room temperature. Other cones are similarly treated for 30 minutes, 2 hours, 4 hours, 8 hours and 16 hours. The treated yarns in each instance are then rinsed and dried, passed over a 9 inch heater strip maintained at 410 to 420° F. and thereafter about a blade edge having a radius of curvature of approximately 35 to 40 microns. The tension in the yarn measured immediately after the yarn passes about the blade edge is from 18 to 22 grams. The yarn is then collected in the form of skeins which are immersed in water at a temperature of 140° F. The yarn in each instance displays a pronounced curly nature when in a tensionless condition and the tendency to curl is not removed by washing the yarn in warm or hot water. While each of the yarns in the above test showed a measure of elasticization, the yarn which was treated for 4 hours was judged superior.

Example II

Example I is repeated except that the time of treatment in each instance is 4 hours and various concentrations (i.e. 0.05 M, 0.1 M, 0.2 M and 0.4 M) of zinc chloride solution are employed. While all yarns treated displayed a measure of elasticization, the yarn treated in 0.1 M zinc chloride was judged to be superior.

Example III

Examples I and II are repeated except that in place of zinc chloride, sulfuric acid solutions of equal molarity are employed. The yarns in this test in each instance displayed a measure of elasticity but the yarn treated in 0.1 M sulfuric acid for a period of 4 hours was the best of the lot.

The procedure for processing yarns of different acetyl content or for processing cellulose ester yarns with other acids or salts is the same as set forth in the above examples.

Having thus described my invention what I desire to claim and secure by Letters Patent is:

1. A process for elasticizing continuous filament cellulose ester yarns which comprises treating the yarn, for a time sufficient to increase its elongation to break but insufficient to severely reduce the strength of the yarn, with an aqueous solution of a compound selected from the group consisting of non-oxidizing inorganic acids, salts thereof with polyvalent metals and mixtures of said salts and said acids, washing the yarn until it is substantially free of solution, heating the yarn to a temperature of at least about 200° F. but not above its sticking temperature, and passing the heated yarn, while under tension of at least about 0.05 gram per denier but insufficient to result in the yarn being severed, about the sharp edge of a blade member in an angular path with said edge disposed at the apex of the angle in the yarn path.

2. A process according to claim 1 wherein the aqueous solution has a concentration of from about 0.05 molar to about 0.4 molar.

3. A process according to claim 2 wherein said compound is zinc chloride.

4. A process according to claim 2 wherein said compound is sulfuric acid.

5. A process according to claim 2 wherein the yarn is cellulose triacetate.

6. A method for elasticizing continuous filament cellulose acetate yarns which comprises treating the yarn in an aqueous solution of zinc chloride for a sufficient period of time to increase its elongation to break but for an insufficient time to severely reduce the strength of the yarn, washing the yarn until it is substantially free of said solution, heating the yarn to a temperature of from about 360° F. to 440° F., and passing the heated yarn, while under a tension of from about 0.05 to 0.4 gram per denier, about the edge of a blade having a radius of curvature of at least about 8 microns but equal to not more than about 3 times the diameter of the yarn.

7. A method according to claim 6 wherein the concentration of the aqueous solution is from about 0.05 to 0.4 molar.

8. A method according to claim 7 wherein the yarn, subsequent to being passed about said blade, is heated while in a substantially tensionless condition.

9. A method according to claim 7 wherein the yarn is cellulose triacetate.

10. A method for elasticizing continuous filament cellulose acetate yarns which comprises treating the yarn in dilute sulfuric acid for a sufficient period of time to increase its elongation to break but for an insufficient time to severely reduce the strength of the yarn, washing the yarn until it is substantially neutral, heating the yarn to a temperature of from about 360° F. to 440° F., and passing the heated yarn, while under a tension of from about 0.05 to 0.4 gram per denier, about the edge of a blade having a radius of curvature of at least about 8 microns but equal to not more than about 3 times the diameter of the yarn.

11. A method according to claim 10 wherein the concentration of said dilute sulfuric acid is from about 0.05 to 0.4 molar.

12. A method according to claim 11 wherein the yarn is cellulose triacetate.

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