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PRODUCTION OF HIGH TENACITY FILAMENTARY MATERIALS

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1 Claim. (Cl. 57-157)

This invention relates to the production of high tenacity filamentary materials, and relates more particularly to an improved process for the saponification of stretched filamentary materials, such as films, threads and yarns (hereinafter referred to as "yarns") having a basis of an organic acid ester of cellulose whereby high tenacity regenerated cellulose yarns are obtained.

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High tenacity regenerated cellulose yarns of high degree of strength have been obtained by 10 in most of the winding and backwinding of bobstretching filamentary materials having a basis of cellulose acetate or other organic acid ester of cellulose from 100 to 2000% of their original length, and then subjecting such stretched yarn to the action of a basic saponifying agent to split 15 off the combined acyl groups.

The saponification of the stretched yarns has been usually effected after the stretched yarns were backwound from the packages, on which they were taken up after the stretching operation, onto a perforated bobbin. A plurality of stainless steel bobbins of yarn were then placed in a suitable enclosed vessel and the saponifying liquid circulated through the windings of yarn on the bobbin by supplying the saponifying liquid 25 thereto under moderate pressure until the saponification of the yarn on the bobbins was completed. This method of saponifying yarn was not wholly satisfactory both from the standpoint of economy and also of manipulation. Thus, the 30 bobbins must preferably be made of stainless steel which made them heavy and expensive. Because of their weight, the bobbins were not only hard to handle but also could not be made desirably large and only a limited amount of yarn 35 could be placed thereon. Moreover, the yarn had to be wound onto the bobbins with extraordinary care otherwise the saponification was effected non-uniformly. Again, since the saponification causes shrinkage of the yarn in diameter 40 my invention may comprise yarns of cellulose by loss of combined acetic acid, often up to 35%, a soft package results making for difficulty in unwinding the same.

Heretofore, the saponification of yarn was also effected while the yarn was in the form of a 45 hank. Hanking, however, is a relatively expensive operation and the manipulating of hanks must be done with great care in order to avoid damage to the yarn. Moreover, the backwinding of hanks is a delicate operation and consumes much labor since tangling the yarn in such yarn packages is very easily effected.

It is, accordingly, an important object of this invention to provide an improved process for

cellulose acetate or other organic acid ester of cellulose which will be free from the foregoing and other disadvantages, and which will produce highly satisfactory high tenacity regenerated cellulose yarn efficiently and economically.

Another object of this invention is the provision of an improved process for the saponification of high tenacity stretched yarns having a basis of an organic acid ester of cellulose, where-

bins is eliminated. Other objects and advantages of this invention

will appear from the following detailed description.

I have found that highly satisfactory high tenacity regenerated cellulose yarns may be produced from stretched yarns having a basis of cellulose acetate or other organic acid ester of cellulose by a process involving a twist-winding 20 of the stretched yarns onto a suitable arbor covered with a fabric sock to form a cake, wrapping the fabric sock completely around the finished cake, removing the cake of yarn from the arbor, saponifying the cake of yarn while covered with the fabric sock, washing and drying the cake of yarn, and then winding the stretched and saponified yarn into the form of the desired ultimate package. It is to be understood, however, that the yarn may first be wound into cake form and the sock placed over said cake after it is removed from the arbor on which it is formed. The regenerated cellulose yarn produced in accordance with my invention is not only more uniform than the products obtained in accordance with prior processes but also has an appreciably higher tenacity. Moreover, there are produced longer lengths of knotless yarn, for the reasons, that larger packages may be processed.

The materials saponified in accordance with acetate or other organic acid ester of cellulose which have been subjected to stretching, in one or a plurality of stages, and taken up on a ring bobbin with a twist of about ½ turn per inch. The stretching may be effected in suitable stretching apparatus with steam or hot water and may be facilitated by means of solvents, latent solvents or swelling agents for the cellulose acetate or other cellulose esters, for example aqueous solu-0 tions of thiocyanates, such as those of the alkali metals, ammonium or alkaline earth metals, or aqueous solutions of zinc chloride; acetic acid, formic acid, lactic acid, diacetone alcohol, acetone, the ethers and esters of ether-esters of saponifying stretched yarns having a basis of 55 olefine and polyolefine glycols, for instance glycol

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mono-acetate, methyl glycol mono-acetate, the acetins, diozane and its homologues and substitution products, methylene ethylene ether and its homologues and substitution products and similar cyclic ethers, dichlorethylene, methylene 5 chloride and the like. Such organic solvents, latent solvents or swelling agents may be applied in conjunction with any suitable diluent, for example, water, benzene and its homologues, petroleum fractions or other aliphatic hydrocarbons 10 and chlorinated hydrocarbons, e.g. carbon tetrachloride, which have precipitating action or at least little or no solvent action upon the cellulose acetate or other organic acid ester of cellulose treated. The stretching for the purposes of the 15 present invention should involve a very substantial extension of the filaments, yarns or other products, and in particular extensions of the order of 100, 200, 300, 500, 1000% or more. The stretching may be applied either to wet or dry 20 free of saponifying agent while still in the spinspun yarns. Instead of or in addition to stretching the yarn after its formation, either continuously or discontinuously with its production, the yarn may be stretched considerably during its formation. In dry spinning operations, this in- 25 volves a softening treatment during the dry spinning process so as to enable substantial stretch to be imparted to the materials, while in the case of wet spinning the nature of the coagulating bath or of the spinning solution may be such 30 that a very considerable stretch may be imparted to the materials during their actual formation. In general, the stretching of the materials, either during, continuously with or after their formation, should be such as to impart thereto a rela- 35 tively high tenacity, preferably in excess of 2 to 2.5 grams per denier, and may attain values of 3 to 4 grams per denier or more. The advantage of stretching to a considerable extent so as to obtain such high tenacities will be appreciated 40 from the fact referred to above that the higher the tenacity of the stretched yarn the greater the increase in tenacity as a result of saponification treatment.

In accordance with my invention, the stretched 45 yarns are then up-twisted into cake form by means of any suitable cake twisting machine such as, for example, a convention twistercheeser adapted to feed the yarn at suitable and controlled tension on which the cake may be 50 formed with a traverse wind at the beginning of the cake of 1 to 11/8. Thus, the stretched yarn from the spinning bobbin on which it was taken up during the stretching operation is up-twisted onto a suitable arbor over which a fabric sock is 55 first drawn, the yarn being cross-wound at the desired density over the fabric sock. The yarn cake is preferably wound on a plastic arbor with an outside diameter of about 5 inches and which is slightly tapered to facilitate removal of the 60 cake therefrom. The cake is preferably wound on the fabric sock covered arbor to an outside diameter of about 71/2 inches with a wall thickness of about 1_{16}^3 inches. After the winding of the cake is removed from the arbor, the sock 65 is wrapped completely around the same and a suitable expander inserted in the yarn cake to maintain the shape thereof. The expander is removed during the subsequent saponification operation and reinserted thereafter remaining 70 into any desired package form employing coninside the cake throughout the remaining operations. The use of the fabric sock offers many advantages; thus, the handling of the cake is effected without damage to the yarn windings, the saponification and subsequent operations are 75 cake is effected,

effected in a more uniform manner resulting in a more uniform product, and the fabric sock surrounding the cake also acts as a filtering medium preventing the passage therethrough of any extraneous substances which may be present in the treating liquids. It is to be understood that the sock may be made of any other suitable material such as, for example, circular knit cotton fabric or a suitably treated paper.

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While the saponification of the yarn cake may be effected in any suitable manner, I have found that optimum results, with respect to uniformity in the resulting product and speed of saponification, are obtained by placing the yarn cake covered with the fabric sock in a rotating perforated spinning bucket and subjecting the yarn cake to the action of a saponifying agent comprising a solution of a basic saponifying agent. The regenerated cellulose yarn produced may be washed ning bucket and in cake form. The saponified yarns are then dried and wound into any desired package form. The regenerated cellulose yarns produced are found to be more uniform, to have higher dry and wet tenacities, to have fewer broken ends and to be cleaner than the regenerated cellulose yarns formed by prior processes of saponifying cellulose acetate or other organic acid esters of cellulose.

The saponifying agent may be in the form of an aqueous, alcoholic or aqueous/alcoholic solution of any suitable basic agent, and may be sprayed onto the stretched cellulose acetate or other organic acid ester of cellulose yarns in any convenient manner. The saponifying agent may be inorganic in nature such as, for example, sodium hydroxide or potassium hydroxide. Organic saponifying agents such as, for example, methyl amine, ethylene diamine or triethanolamine, may also be employed. It is preferred that the saponifying solution also contain from about 6 to 10% by weight of sodium acetate which assists materially in preventing degradation of the yarn undergoing saponification. Preferably, I employ an aqueous solution of sodium hydroxide as the saponifying agent and in concentrations of 0.1 to 5% by weight. I have obtained highly desirable results by employing a 0.6% aqueous solution of sodium hydroxide containing 8 to 10% of sodium acetate as the saponifying medium and supplying this medium to the spinning bucket at a temperature from 140 to 180° F. and at a rate of 4 gallons per minute. While the temperature of the saponification medium may range from 20 to 100° C., I have found that optimum results are obtained when the temperature of the saponification medium is as stated, namely from 140 to 180° F.

The saponification medium is introduced in the center of the cake in the spinning bucket and centrifugally distributed and dispelled throughout the cake. The spinning bucket is preferably rotated at the rate of about 730 R. P. M. The cake of yarn is subjected to the saponification treatment rotating spinning bucket for about 2 hours after which it is washed while still in the spinning bucket and finally dried for 24 hours at a temperature of about 150 to 180° F. The cake is then placed on a wood cake holder and wound ventional textile apparatus.

In the drawing:

Fig. 1 is a view, partly in section, showing a centrifuge in which the saponification of the yarn

Fig. 2 is a perspective view of the yarn cake surrounding the fabric sock as it is taken off the arbor on which the yarn cake is wound, and

Fig. 3 is a cross-sectional view of the yarn cake completely enclosed by the fabric sock.

Like reference numerals indicate like parts throughout the several views of the drawing.

Referring to the drawing, the reference numeral 4 indicates a tank for holding the saponifying agent which is fed through conduit 5 to a 10 pump 6 adapted to supply the saponifying agent at the desired rate to a centrifuge generally indicated by reference numeral 7 through a conduit 8. The lower end of conduit 8 is perforated and forms a distributing header 9 extending within 15 the centrifuge.

The centrifuge 7 comprises a spinning pump 11 to which is attached a shaft 12 extending through the bottom of the outer housing 13 of the centrifuge. The shaft 12 is adapted to be rotated at the desired speed by any suitable means. The spinning bucket is provided with openings 14 in the centrifugal wall thereof in which the saponifying agent is removed as it passes through the provided with a suitable drain or outlet 16 which, if desired, may be connected to a pipe 17 for returning the saponifying agent to the tank 4 for recycling the same.

The yarn cake 15 which was wound on an arbor 30 with a fabric sock 18 has the sock completely covering the same with the ends overlapping, as shown in 19 in Fig. 3, before it is placed in the spinning bucket 11 of the centrifuge. The yarn cake is placed in the spinning bucket in such a 35 cellulose employed may be of any degree of middle of the opening in said yarn cake.

If desired, an annular plate 21 is placed upon the yarn cake, remaining thereon throughout the saponification process. The weight of the annular plate 21 is such that it will cause contraction in the yarn cake as the saponification of said yarn cake proceeds causing the denier of the yarn forming the cake to decrease.

In order to further illustrate my invention but 45 to secure by Letters Patent is: without being limited thereto the following example is given:

Example

A 40 filament, 32.5 denier cellulose acetate yarn 50 which has been given a stretch of 900% is backwound with a twist of 21/2 turns per inch, from the bobbins on which the yarn was taken up with a twist of 1/2 turn per inch during the stretching operation, in the form of a cake on to the 55 arbor of a Whitin twister cheeser, the arbor being slightly tapered and being first covered with a fabric sock. The yarn cake is wound on the fabric sock to a weight of 1.7 pounds with a traverse wind of 1 to 1% at the start 60 of the cake. The yarn cake and the fabric sock are then removed together from the arbor, the ends of the sock are then turned on the outer windings of the yarn cake so as to cover the yarn cake completely and an expander is 65 inserted in the covered yarn cake to maintain the shape thereof.

The fabric sock-covered yarn cake, after the expander is removed therefrom, is placed in a spinning bucket and a centrifuge and an an- 70 M nular plate of appreciable weight is seated thereon. Saponifying liquid comprising a 0.6% aqueous solution is forced into a spinning bucket at a speed of 730 R. P. M. at the rate of 4 gallons per minute for 2 hours, the temperature of the 75

saponifying liquid being 140° F. The saponified yarn cake is then washed for 30 minutes with softener-heated water at 140° F. and dried for 24 hours at 150° F. The expander is again inserted in the dried yarn cake and yarn of the cake is then wound into a cone. The regenerated cellulose yarn produced by this economical method is of better quality with respect to uniformity of saponification than the regenerated cellulose produced by saponifying stretched yarn by the bobbin method. The regenerated cellulose yarn produced in accordance with my process has a dry tenacity of 7.12, a dry elongation of 6.3, a wet tenacity of 6.20 and wet elongation of 6.5.

The cellulose acetate yarns stretched and saponified in accordance with the process of this invention to yield regenerated cellulose of improved quality may be prepared by extruding a solution of cellulose acetate dissolved in a suitable solvent through a plurality of orifices into an evaporative medium as in the dry method of spinning or into a coagulating or precipitating liquid medium as in the wet method of spinning. yarn cake 15. The bottom of outer casing 13 is 25 to form the desired multi-filament yarn which The filaments so formed are associated together are first stretched and then saponified. In addition to cellulose acetate, organic acid esters of cellulose may be employed for preparing regenerated cellulose yarns of improved tenacity. Such other organic acid esters of cellulose may be cellulose propionate, cellulose butyrate and mixed esters of cellulose, such as cellulose acetatepropionate and cellulose acetate-butyrate. The cellulose acetate or other organic acid ester of esterification, for example, where cellulose acetate is employed it may have an acetyl value of from 40 to 62.5%, calculated as acetic acid.

It is to be understood that the foregoing de-40 tailed 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

In a process for preparing regenerated cellulose yarn of increased tenacity, the steps of winding stretched yarn having a basis of an organic acid ester of cellulose into cake form on a fabric stock while inserting a twist in said stretched yarn, completely covering said yarn cake with said fabric sock, placing said fabric covered varn cake in a vessel rotating at a speed of 730 R. P. M., feeding a saponifying medium comprising a 0.6% aqueous solution of sodium hydroxide, containing 8 to 10% of sodium acetate, at a temperature of 140° F. into the rotating vessel at a rate of 4 gallons per minute to convert said organic acid ester of cellulose yarn into regenerated cellulose yarn, and winding said regenerated cellulose yarn from cake form into another form of yarn package.

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The following references are of record in the file of this patent:

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Certificate of Correction

May 9, 1950

Patent No. 2,506,710

CAMILLE DREYFUS

It is hereby certified that errors appear in the printed specification of the above

Column 6, line 3, for "softener-heated" read softener-treated; line 50, for the word "stock" read sock; numbered patent requiring correction as follows:

and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office. Signed and sealed this 22nd day of August, A. D. 1950.

[SEAL]

THOMAS F. MURPHY, Assistant Commissioner of Patents.