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METHOD OF PRODUCING HIGH-TENACITY REGENERATED CELLULOSE YARNS

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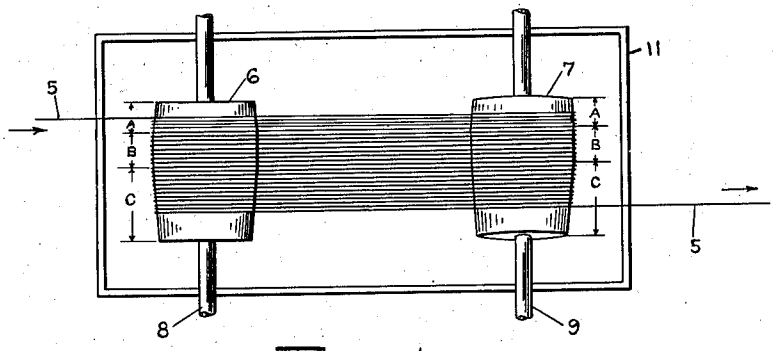


Fig-1

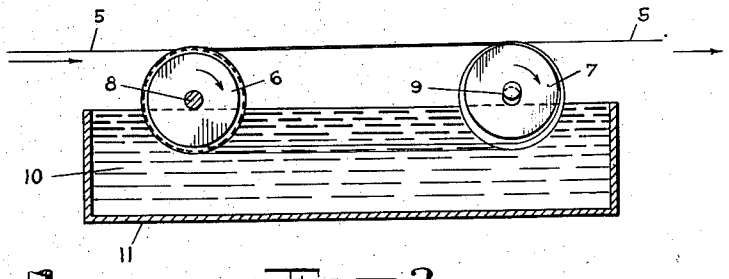


Fig-2

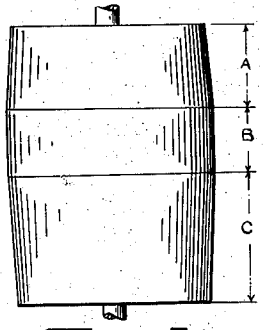


Fig-3

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METHOD OF PRODUCING HIGH TENACITY REGENERATED CELLULOSE YARNS

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This invention relates to the preparation of high tenacity regenerated cellulose yarns, and relates more particularly to improved saponification methods for the production of said high tenacity regenerated cellulose yarns from stretched yarns having a basis of cellulose acetate, or other organic ester of cellulose.

An object of this invention is the preparation of regenerated cellulose yarns of higher tenacities by a process involving the saponification of stretched yarns having a basis of cellulose acetate or other organic ester of cellulose.

Another object of this invention is the provision of an improved saponification process for the production of said regenerated cellulose yarns from stretched yarns of cellulose acetate or other organic ester of cellulose wherein higher yarn tenacities with limited weight loss of the saponified yarns are obtained.

A further object of this invention is the provision of improved apparatus for the continuous saponification of yarns, and more especially, stretched yarns having a basis of cellulose acetate or other organic ester of cellulose, wherein means are provided for controlling the tension exerted thereon.

Other objects of this invention will appear from the following detailed description and the accompanying drawing.

In the drawing, wherein a preferred embodiment of our novel apparatus for carrying out our invention is shown,

Fig. 1 is a plan view of an improved apparatus for the continuous, controlled saponification of cellulose acetate or other organic ester of cellulose yarns,

Fig. 2 is a side elevational view, partly in section, of this apparatus showing the roller system in askewed relation, and

Fig. 3 is a plan view, on an enlarged scale of one of the rollers employed in our novel continuous saponification apparatus.

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

Yarns of regenerated cellulose of high tenacity have been produced by the saponification of stretched yarns having a basis of an organic ester of cellulose, such as, for example, cellulose acetate. The stretching of the yarns is effected during some stage in their manufacture and the yarns may be increased in length as a result of the stretching operation to the extent of 200, 300, 500, or even 1000 or 2000% of their original length. The yarns acquire a relatively high tenacity on being stretched, the tenacity being retained and in many instances enhanced by saponification

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which, as is well known, effects a regeneration of the cellulose. The resulting high tenacity regenerated cellulose yarns are of great strength and find extensive employment where yarns of an unusual degree of strength and dimensional stability are desired.

During the saponification of the stretched cellulose acetate or other organic ester of cellulose yarns, the latter undergo various physical and chemical changes. One important physical change which the stretched cellulose acetate yarns undergo during saponification is an increase in length at the start of saponification due to linear as well as lateral swelling of the yarns as they are wet out followed by marked linear shrinkage involving an appreciable decrease in length. The shrinkage varies depending upon the nature of the stretched product undergoing saponification. A considerable loss in the weight of the cellulose acetate yarns also takes place due to the fact that the acetyl groups present are split off by the saponifying agent employed. Moreover, this chemical change causes the yarns to undergo a pronounced decrease in denier which, because the breaking load is usually retained, produces yarns of substantially increased tenacity. An additional weight loss also takes place by reason of the fact that certain alkali-soluble fractions of the regenerated cellulose produced are removed as well. Such weight and shrinkage losses decrease the yield of regenerated cellulose yarn which may be obtained from a given quantity of stretched cellulose acetate yarns which decrease is, of course, undesirable.

We have now discovered that regenerated cellulose yarns of superior physical characteristics may be obtained with a minimum weight loss by an improved saponification process. In accordance with our novel saponification process, the stretched cellulose acetate or other organic ester of cellulose yarns are saponified under a predetermined and controlled tension so that the ultimate shrinkage effected during saponification is held within predetermined limits, whereby the shrinkage effected as a result of the saponification reaction is less than that normally produced when such yarns are saponified in a relaxed condition, e. g. in the absence of tension, and allowed to shrink freely. Surprisingly enough, this novel method of saponifying cellulose acetate or other organic ester of cellulose yarns, wherein the yarns are maintained under controlled tension during saponification, produces substantially completely regenerated cellulose yarns of an even higher tenacity then are normally obtained with the same cellulose acetate yarns, but saponified in

a relaxed condition. Furthermore, we have found that by our novel saponification process not only is the weight loss of the yarns due to saponification less than that usually produced but also that the regenerated cellulose yarns obtained are of increased softness and pliability. It is believed that our novel tension saponification process prevents certain of the cellulosic fractions which are formed on saponification from being extracted by the saponifying bath and that the retention of these cellulosic fractions in the regenerated cellulose yarns obtained is responsible for the improved pliability and softness possessed by the yarns obtained in accordance with our process. This explanation, however, is merely a theoretical one and we do not wish to be bound thereby.

The desired degree of control over the shrinkage of the cellulose acetate yarns is achieved by maintaining the yarns under predetermined tension during the progress of the saponification reaction, which tension may be obtained in any convenient manner. Thus, for example, the yarns may be saponified in a continuous manner while passing from one yarn guide to another in the saponifying bath employed, the yarn guides being spring loaded or otherwise adapted to maintain the yarns under the desired degree of tension at each stage of the saponification.

Preferably, however, the yarns undergoing saponification are maintained under the desired controlled tension by being passed continuously from one driven roller to another, partially or wholly submerged in the desired saponifying bath. To this end we employ a novel apparatus comprising a plurality of rollers the contour of which is such as to permit some shinkage of the yarns to take place during the saponification process, but at the same time to maintain the yarns under a continuous and predetermined amount of tension at each stage of the saponification operation, whereby the total degree of shrinkage taking place is limited, and is less than that effected when the yarns are saponified in a completely relaxed condition.

Referring now to the drawing, a stretched cellulose acetate yarn 5 coming from a suitable source of supply (not shown) is passed, as many times as desired, around a pair of identical rollers 6 and 7 mounted on shafts 8 and 9 respectively, the shaft 9 carrying roller 7 being somewhat askew with respect to shaft 8. The rollers 6 and 7 are shown on a somewhat enlarged scale in Fig. 3. Shafts 8 and 9 on which the rollers are mounted are provided with suitable driving means (not shown) so that they may be driven at any desired speed, thus continuously advancing yarn 5 through the bath. The method of mounting shafts 8 and 9 so that shaft 9 is not parallel to shaft 8, gives a lead to the yarn passing around said rollers and not only keeps the yarn wraps apart but also causes yarn 5 to move laterally along the surface of the rollers. Rollers 6 and 7 are partially submerged in a saponifying bath 10 of any suitable composition which is contained in a trough 11. As stated, the rollers 6 and 7 may, if desired, also be wholly submerged in bath 10, thus maintaining the cellulose acetate yarn 5 in constant contact with the saponifying bath 10.

The initial effect of the saponifying bath 10 is to wet out yarn 5. This action tends to relax the stretched yarn 5 somewhat, permitting it to increase in length. Any sagging, however, is avoided by gradually increasing the tension on the yarn to compensate for any increase in length. This take-up is effected by the lateral movement

of yarn 5 across the sections of rollers 6 and 7 generally indicated by the letter A. The sections A are of gradually increasing diameter, and therefore act to take up any increase in the length due to relaxation of the yarn. As the saponifying action of bath 10 begins to effect yarn 5, the latter gradually begins to shrink. The rollers 6 and 7, however, are rotated at such speed in timed relation to the rate of saponification that when this phase of the saponification is reached the yarn 5 is passing over section B of said rollers. This section is not tapered but is substantially cylindrical in form since while passing over section B the rate of shrinkage is not sufficient to require a change in the contour of the roller to eliminate the excessive development of tension due to shrinkage. However, since the yarn continues to shrink as it undergoes further saponification, an increasing degree of tension is placed upon the yarn due to the unyielding rollers. Accordingly, as saponification proceeds further, the yarn undergoing saponification gradually moves in a lateral direction across the contoured rollers 6 and 7, still in timed relationship to the degree of saponification, and finally reaches section C of said rollers which section is provided with a suitable taper. The taper of section C is such that it enables the yarn to shrink but at the same time maintains it under the desired tension. The contour of section C is such as to compensate for a gradually increasing rate of shrinkage and this prevents the tension on the yarn from building up to an undesirably high level. The substantially completely saponified yarn leaving roller 7 may be washed, neutralized and dried in a continuous manner, or the yarn may be taken up in the form of a suitable package and the yarn may be neutralized, washed and dried in package form. During drying, the yarn may be allowed to shrink freely. The degree of shrinkage produced as a result of drying will vary with the extent to which the saponified yarns are dried. The lower the moisture content to which said yarns are dried the greater will be the degree of shrinkage effected during drying.

The tensions under which the yarns are maintained during the stages of the saponification operation whereby the degree of shrinkage they undergo is limited, may be from 0.02 to 0.5 gram per denier but optimum results are achieved when the yarns undergoing saponification are maintained under tensions of about 0.03 gram per denier.

The saponification may be effected by means of any suitable basic agent. The saponifying agent may be inorganic in nature such as, for example, sodium hydroxide, potassium hydroxide, sodium silicate, ammonium hydroxide, sodium carbonate or other inorganic basic agents. Organic saponifying agents may also be employed such as, for example, methylamine, ethylene diamine, triethanolamine, or other organic bases. The saponifying agent may be employed in varying concentrations in aqueous, alcoholic, or aqueous-alcoholic solutions. Advantageously, the saponifying bath may be at a temperature of 20 to 100° C. during the saponification process. For example, when aqueous solutions of sodium hydroxide are employed as the saponifying agent, the sodium hydroxide may be present in a concentration of from 0.1 to 5% by weight and saponification may be effected in from 1 to 60 minutes with the solution at a temperature of from 30 to 90° C. When employing aqueous solutions of sodium hydroxide as the saponifying

medium, it is preferable to include a salt such as sodium sulfate or sodium acetate in said solution.

To effect the desired saponification the stretched yarns are led directly into the saponifying bath, the path through the saponifying bath being sufficiently extended and the speed at which the yarns pass through the bath being such that the yarn, under the conditions of both temperature and concentration employed, is completely saponified before leaving the bath. Speeds of 10 to 100 meters per minute are satisfactory.

While our invention has been more particularly described in connection with continuous saponification processes, batch processes may also be employed for saponifying the cellulose acetate yarns under tension employing suitable yarn holders for maintaining the yarns under tension.

In order further to illustrate our invention, but without being limited thereto the following example is given:

Example

A 144 denier, 120 filament cellulose acetate yarn, which has been prepared by subjecting a 1440 denier yarn to a 10 times stretch, is saponified in a continuous manner at 80° C. with a saponifying bath comprising a 0.7% aqueous solution of sodium hydroxide containing 8.5% by weight of sodium acetate. The yarns are passed through the saponifying bath at a speed of 10 meters per minute and the path of the yarn is of such extent that it is subjected to the action of the saponifying bath for 11 minutes. During saponification the yarns passing through the bath are maintained under a continuous tension in the manner heretofore described, employing a pair of driven, novel contoured rollers around which the running yarn is passed. The initial taper of the rollers is such that as the yarn passes into the bath it is placed under tension with the taper of the rollers extending the yarn path to avoid the formation of any slack because of the relaxing or swelling effect of the initial wetting. The slackening permits the yarn to increase about 0.35% in length which is, of course, taken up the increasing diameter of the contoured rollers. The tension under which the yarn is maintained amounts to about 0.03 gram per denier. The loops are then led across the tapered rollers in timed relation to the shrinkage and to the saponification. The contour of the rollers is such that the yarns are also maintained under a substantially uniform tension of 0.03 gram per denier during the initial saponification across the flat portion of the rollers as well as during the latter stage of saponification across the final tapered portion. The degree of shrinkage permitted by maintaining the yarns under tension is thereby limited to about 0.35% of the original yarn length, a total change in length of 0.70% taking place when determined from the initial, relaxed state. The saponified, regenerated cellulose yarns obtained are washed, neutralized with a 1% aqueous solution of acetic acid, washed and dried at 80° C. to a moisture content of 0.75%. The regenerated cellulose yarn produced is of about 100 denier, has a dry tenacity of 7.3 grams per denier and a wet tenacity of 6.5 grams per denier. The over-all linear shrinkage of the yarn from its original length is found to be only 0.9%, a shrinkage of 0.55% having occurred during drying to the particular moisture content indicated. When allowed to shrink freely during

like saponification, said yarn shrinks about 0.55% in length due to the saponification and a total of 1.45% when dried to a like moisture content, a shrinkage of 0.9% taking place as a result of drying. The weight loss of the yarns saponified under tension in accordance with our novel process amounts to about 37.5% while the weight loss for like cellulose acetate yarns saponified under the same conditions but in a relaxed state amounts to about 40%.

The cellulose acetate or other organic ester of cellulose yarns which may be stretched and then saponified in accordance with our novel process to yield regenerated cellulose yarns of improved tenacity may be prepared by extruding a solution of cellulose acetate or other organic ester of cellulose dissolved in a suitable volatile 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. The filaments formed in this manner are associated together to form the desired multi-filament yarn or thread which is first stretched and then saponified. In addition to cellulose acetate, other organic esters of cellulose which may be employed for the preparation of the yarns are, for example, cellulose propionate, cellulose butyrate, and mixed esters such as, cellulose acetate-propionate and cellulose acetate-butyrate.

The cellulose acetate or other organic ester of cellulose employed may be of any degree of esterification. For example, in the case of cellulose acetate materials, they may have an acetyl value of from 40 to 62%, calculated as acetic acid.

After the yarn or thread is formed, it is then stretched. This is usually done when the yarn is in a somewhat softened condition. The yarn may be softened by treating it with a solvent, latent solvent, or swelling agent for the cellulose acetate or other organic ester of cellulose. This softening treatment may be carried out by bath or padding methods, or by exposing the cellulose acetate materials to vapors of said organic solvents, latents solvents or swelling agents during, or just prior to, the stretching operation. The yarns may also be softened by radiant heat or by the use of steam or hot water. After softening, the yarns may be stretched in a continuous manner by being passed from a roll which rotates at a slow speed to one rotating at a higher speed, the roller rotating at a higher speed serving to give the yarn the desired stretch. The degree of stretch may be varied by increasing or decreasing the differential in the speeds of the rollers. The yarn may be stretched in a plurality of stages, if desired. For example, part of the stretch may be imparted to the yarn by softening and stretching in one stage and the remainder of the stretch may be imparted by a second softening and stretching operation, but more than two stages of stretch may, of course, be employed. After stretching the yarns may be taken up on bobbins or may be collected in a centrifugal spinning box or Topham box and are ready for saponification in accordance with our novel process.

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

Having described our invention, what we desire to secure by Letters Patent is:

1. In a process for the production of improved high tenacity regenerated cellulose yarns by the

saponification of stretched yarns having a basis of an organic ester of cellulose, wherein the yarns undergoing saponification increase in length and then decrease in length as said saponification proceeds, the step which comprises continuously feeding said organic ester of cellulose yarns into a saponifying bath and placing the wetted yarns under a controlled and predetermined tension of 0.02 to 0.5 gram per denier in the saponifying bath throughout the entire saponification process, whereby the degree of shrinkage effected by the saponification reaction is limited to less than that effected when said yarns are saponified in a substantially untensioned condition.

2. In a process for the production of improved high tenacity regenerated cellulose yarns by the saponification of stretched yarns having a basis of cellulose acetate, wherein the yarns undergoing saponification increase in length and then decrease in length as said saponification proceeds, the step which comprises continuously feeding said cellulose acetate yarns into a saponifying bath and placing the wetted yarns under a controlled and predetermined tension of 0.02 to 0.5 gram per denier in the saponifying bath throughout the entire saponification process, whereby the degree of shrinkage effected by the saponification reaction is limited to less than that effected when said yarns are saponified in a substantially untensioned condition.

3. In a process for the production of improved high tenacity regenerated cellulose yarns by the saponification of stretched yarns having a basis of cellulose acetate, wherein the yarns undergoing saponification increase in length and then decrease in length as said saponification proceeds, the step which comprises continuously feeding said cellulose acetate yarns into a saponifying bath comprising an aqueous alkaline medium and placing the wetted yarns under a controlled and predetermined tension of 0.02 to 0.5 gram per denier in the saponifying bath throughout the entire saponification process, whereby the degree

of shrinkage effected by the saponification reaction is limited to less than that effected when said yarns are saponified in a substantially untensioned condition.

4. In a process for the production of improved high tenacity regenerated cellulose yarns by the saponification of stretched yarns having a basis of cellulose acetate, wherein the yarns undergoing saponification increase in length and then decrease in length as said saponification proceeds, the step which comprises continuously feeding said cellulose acetate yarns into a saponifying bath comprising an aqueous solution of sodium hydroxide and placing the wetted yarns under a controlled and predetermined tension of 0.02 to 0.5 gram per denier in the saponifying bath throughout the entire saponification process, whereby the degree of shrinkage effected by the saponification reaction is limited to less than that effected when said yarns are seponified in a substantially untensioned condition.

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