

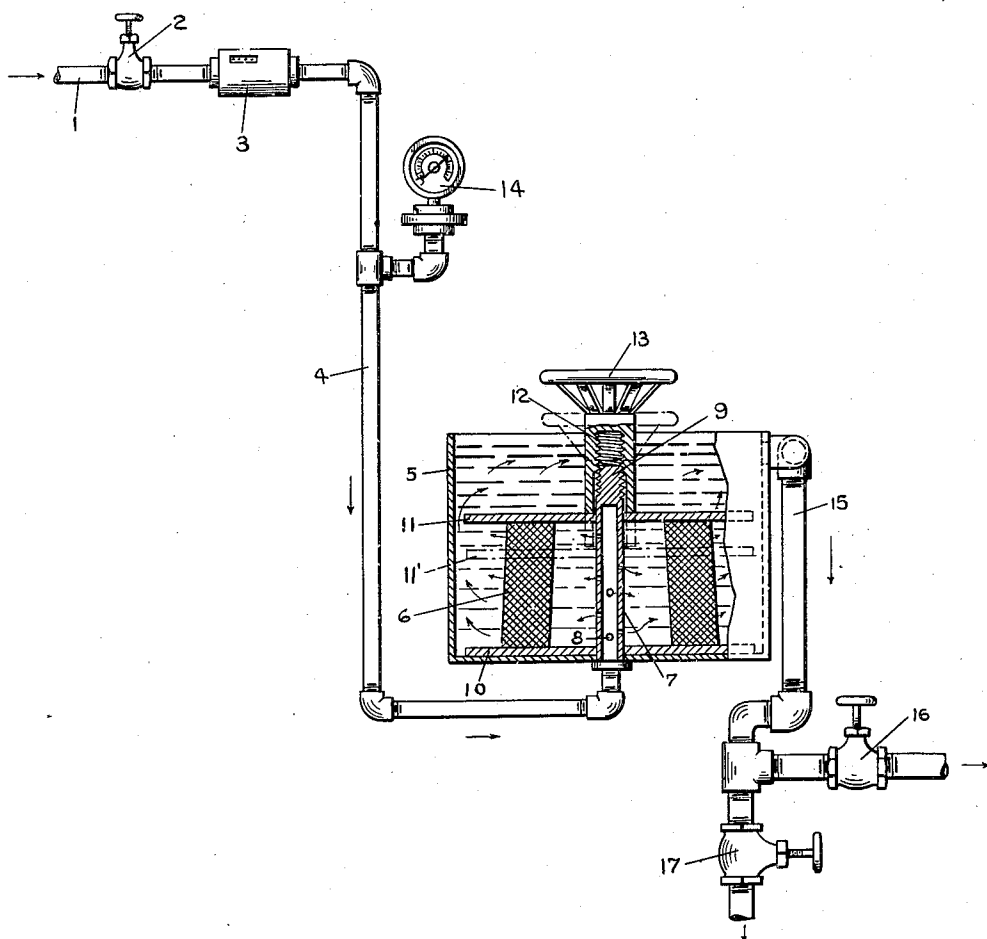
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PROCESS OF SAPONIFYING YARN CAKES

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PROCESS OF SAPONIFYING YARN CAKES

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This invention relates to the treatment of textile material, and relates more particularly to the production of high tenacity regenerated cellulose yarns by the saponification in cake form of stretched or unstretched yarns having a basis of an organic acid ester of cellulose. This invention also relates to improved apparatus whereby said saponification process may be effected.

An object of this invention is the provision of an improved process for the saponification, in cake form, of stretched or unstretched yarns having a basis of an organic acid ester of cellulose whereby completely saponified high tenacity regenerated cellulose yarns of improved physical characteristics are obtained.

Another object of this invention is to provide a novel and efficient apparatus for effecting the saponification of stretched or unstretched organic acid ester of cellulose yarns in cake form whereby complete saponification may be achieved in a relatively short time with the production of a cake of high tenacity regenerated cellulose yarn which retains the original relation of the yarn windings and is readily amenable to washing, winding, coning and like processing.

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

In the drawing, the figure is a front elevational view, partly in section, of our novel apparatus for effecting the saponification of organic acid ester of cellulose yarns in cake form.

In the production of high tenacity regenerated cellulose yarns by the saponification of stretched or unstretched yarns having a basis of cellulose acetate or other organic acid ester of cellulose, the yarns are usually saponified in package form. The yarns are taken up on bobbins, the bobbins placed in a closed vessel into which a suitable saponifying medium is entered and the yarn-filled bobbins maintained therein at a given temperature for a sufficient time to saponify the yarns completely. This saponifying operation not only requires the handling of a large number of relatively small yarn packages but is time-consuming as well. Furthermore, since the yarn undergoes a substantial decrease in denier as a result of the action of the saponifying medium, which acts to split off combined acyl groups, the yarn packages of regenerated cellulose yarn which are produced are quite soft. The softness of the wind makes subsequent rewinding or other processing of the regenerated cellulose yarns difficult. The saponification of cellulose acetate or other organic acid ester of cellulose yarns in a more rapid manner wherein the high tenacity regenerated cellulose

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yarns produced are obtained in a package form of sufficient density to allow the yarns to be readily subjected to further processing has long been the desideratum in the production of these saponified yarns.

We have now found the stretched or unstretched yarns having a basis of cellulose acetate or other organic acid ester of cellulose may be saponified rapidly and completely and high tenacity regenerated cellulose yarns of superior physical characteristics obtained in firm and easily handled package form. In accordance with the novel process of our invention, these improved regenerated cellulose yarns may be obtained in such package form by taking up said cellulose acetate or other organic ester of cellulose yarns in a rotating spinning bucket or Topham box, as employed in the viscose art, whereby a relatively large yarn cake is formed and saponifying the yarn while in cake form and free of an internal yarn holder by forcing a liquid saponifying medium through the yarn windings under pressure and intermittently compressing the yarn cake in a direction along the winding axis as saponification proceeds. Preferably, the liquid flow is halted, while effecting each separate compression of the yarn cake. By our novel process, complete and uniform saponification is effected in a very short time without any channeling of the liquid saponifying medium through the yarn windings and the saponified cake of high tenacity regenerated yarns produced is obtained in a firm, dense, compact form with the continuity of the original wind substantially wholly retained. The retention of the original continuity of the wind and the compact form in which the saponified yarn cake is produced enables the yarn cake to be handled with ease and washing, lubricating, back winding, coning or other processing operations may be carried out rapidly and efficiently.

The saponifying medium forced through the yarn cake may be an aqueous, alcoholic or aqueous/alcoholic solution of any suitable basic saponifying 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 agent. Organic saponifying agents may also be employed, such as, for example, methyl amine, ethylene diamine, triethanolamine or other organic bases. The temperature of the solution of the saponifying agent applied to the yarns may vary, with temperatures of from 20 to 100° C. being satisfactory. Preferably, in saponifying stretched cellulose acetate yarns, we employ aqueous solutions of sodium hydroxide as

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the saponifying agent and in concentrations of 0.1 to 5% by weight, the solution being applied to the yarns undergoing saponification at a temperature of from 20 to 90° C. Optimum results are achieved employing an 0.2 to 2% aqueous solution of sodium hydroxide as the saponifying agent, the solution being maintained at about 80° C. during saponification. Preferably, the solution should also contain from about 5 to 20% by weight of sodium acetate or sodium sulfate.

When saponifying said cellulose acetate yarns in the form of a cake of 1 to 3 pounds in weight, the flow of saponifying solution through the yarn windings may be at a rate of $\frac{1}{2}$ to 3 gallons per minute, the pressure on the yarn cake being such as to cause the liquid flowing through the yarn cake to be maintained at a back pressure of 2 up to 10 pounds per square inch. Complete saponification of the cake of stretched cellulose acetate yarn may be affected under these conditions in from 20 to 60 minutes. After saponification is completed, the cake of high tenacity regenerated cellulose yarns obtained may be washed neutral with water, lubricated and dried.

The yarns forming the yarn cake are completely and uniformly saponified and, as stated, the yarn cake produced retains the original structural relation of the initial windings. The cake may be back wound very readily and the yarn taken up in the form of any desired yarn package without danger of tangling or other physical breakdown in the structure of the yarn cake due to derangement of the yarn windings.

Referring now to the drawing in which a preferred embodiment of the apparatus employed in carrying out our novel process is shown, the liquid saponifying medium employed at the desired temperature and concentration, is forced into the system through a pipe 1 provided with a valve 2 to regulate the volume of flow as well as the pressure and then through a flowmeter, which is indicated by reference numeral 3. The liquid leaving flowmeter 3 passes downward through a pipe 4 and then enters an open chamber 5 containing the yarn cake 6. The liquid enters chamber 5 through a distribution header 7 set inside yarn cake 6, the header 7 being provided with a plurality of discharge ports 8. The upper part of header 7 is blocked off and is threaded at 9.

Yarn cake 6 in chamber 5 is set on a circular plate 10 and covered with a second circular plate 11, parallel to plate 10, the weight of upper plate 11 normally serving to keep it seated on yarn cake 6. However, to maintain plate 11 in position on yarn cake 6, as the back pressure of the liquid forced through the yarn cake tends to unseat it, a stop or control arrangement is provided. This stop mechanism comprises an internally threaded sleeve 12 carrying a circular handle 13, integral therewith, the whole being set on the upper threaded portion 9 of distributing head 7 so that it may be brought to bear on plate 11.

At the start of the saponification, sleeve 12 is rotated by handle 13 in the direction necessary to bring the base of the sleeve to bear on the upper surface of plate 11, only sufficient pressure being exerted to maintain an effective liquid seal for the liquid on the inside of the cake and to maintain the desired liquid pressure. The liquid pressure is indicated on a pressure gauge 14 set in the system and communicating, for example, with pipe 4. As the saponification of yarn cake 6 proceeds, the denier of the yarn forming the cake decreases, which tends to open the wind, due to the decrease in package density. This action al-

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lows the saponifying liquid to flow more rapidly through the windings and the back pressure of the liquid therefore, slowly drops, the drop in pressure being shown on gauge 14. Since the saponifying liquid will tend to channel through the cake when there is a sizeable decrease in the density of the yarn windings, the position of plate 11 is reset manually to avoid channeling as well as to increase the density of the yarn cake 6. The resetting is done by closing valve 2 thereby stopping the flow of saponifying liquid, allowing plate 11 to settle to a lower level and then rotating sleeve 12 by means of handle 13 causing said sleeve to move downward and hold plate 11 in the new setting. Having thus increased the density of the yarn windings to a degree sufficient to maintain the desired liquid back pressure, channeling of the cake is prevented. Saponification is then continued by opening valve 2 and permitting the flow of the saponifying liquid to continue. The operation of resetting plate 11 is repeated periodically as saponification proceeds, the flow of liquid being stopped prior to resetting plate 11 and started again after plate 11 has settled down to the new position.

At the completion of saponification, plate 11 will usually be positioned at a point appreciably lower than its starting position, this point being shown at 11'. The height of the yarn cake is normally reduced about 25 to 40% during the saponification. The spent saponifying liquid is discharged from vessel 5 through a pipe 15 set near the top, or near the bottom if desired and may be returned for further use in the process through valve 16 or discharged to waste by closing valve 16 and opening valve 17.

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

Example

A cake of stretched cellulose acetate yarn obtained by collecting 500 grams of a two-ply 144 filament 120 denier cellulose acetate yarn which has been given a 10 time stretch, is placed in our novel saponifying device between circular plates 10 and 11 with the taper of the yarn cake in an upward direction. If desired, the yarn cake may be wrapped in a cloth sock of the type normally employed in the art. The saponifying liquid, comprising an 0.6% aqueous solution of sodium hydroxide containing 6 to 7 % of sodium acetate, is forced into the system and through the yarn cake 6 at a rate of 2.5 gallons per minute, the solution being at a temperature of 80° C.

At the start of the operation the back pressure is set at about 3.5 pounds per square inch gauge. After five minutes, the pressure drops to 2.5 pounds per square inch due to the fact that the density of the yarn cake has decreased because the action of the saponifying liquid is causing a decrease in the denier of the yarns. The liquid flow is stopped at this point and the upper plate 11 is allowed to settle, dropping about $\frac{1}{8}$ of an inch, and sleeve 12 is then rotated sufficiently to hold lower plate 11 at the new position. Valve 2 is opened and the liquid flow is resumed. The back pressure indicated by gauge 14 is now $3\frac{3}{4}$ pounds per square inch. The pressure drops to 3 pounds per square inch after another five minute intervals, the flow of liquid is stopped and plate 11 is permitted to reset. The resetting operation is continued at approximately five minute intervals for a total saponification period of 1 hour, the liquid flow being cut off at each resetting of plate 11. At this point the back

pressure is about 6.5 pounds per square inch due to the increased density of the yarn cake windings. At the end of the saponification treatment, the upper plate has moved downward a total of 1.5 inches. The yarn cake obtained, which now comprises a firm, coherent, easily handled cake of completely regenerated cellulose yarns, is washed with water at 50° C. for 15 minutes at a flow rate of 2.5 gallons per minute. The cake is then dried and may be back wound to any desired package form. The regenerated cellulose yarns forming the cake have an average dry tenacity of 7.22 grams per denier and a wet tenacity of 6.25 grams per denier.

The cellulose acetate or other organic acid 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 acid 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, or mixed cellulose ether-esters such as ethyl cellulose acetate.

The cellulose acetate or other organic acid 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 62.5%, calculated as acetic acid.

After the yarn or thread is formed, it is then stretched where especially high tenacity yarns are desired. This is usually done while 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, latent 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 yarn may be stretched in a continuous manner by being passed from a roll which rotates at a low 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 are collected in a centrifugal spinning box or Topham box and the yarn cake formed is 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 organic acid ester of cellulose yarns in cake form employing a liquid saponifying medium, the steps which comprise forcing the liquid saponifying medium under pressure through the windings of the yarn cake and intermittently compressing the yarn cake as saponification proceeds and the back pressure exerted by the liquid saponifying medium drops, the liquid flow being halted each time said cake is compressed.

2. In a process for the production of improved high tenacity regenerated cellulose yarns by the saponification of cellulose acetate yarns in cake form employing a liquid saponifying medium, the steps which comprise forcing the liquid saponifying medium under pressure through the windings of the yarn cake and intermittently compressing the yarn cake as saponification proceeds and the back pressure exerted by the liquid saponifying medium drops, the liquid flow being halted each time said cake is compressed.

3. In a process for the production of improved high tenacity regenerated cellulose yarns by the saponification of cellulose acetate yarns in cake form employing a liquid saponifying medium comprising an aqueous solution of sodium hydroxide, the steps which comprise forcing the liquid saponifying medium under pressure through the windings of the yarn cake and intermittently compressing the yarn cake as saponification proceeds and the back pressure exerted by the liquid saponifying medium drops, the liquid flow being halted each time said cake is compressed.

4. In a process for the production of improved high tenacity regenerated cellulose yarns by the saponification of organic acid ester of cellulose yarns in cake form employing a liquid saponifying medium, the steps which comprise forcing the liquid saponifying medium under pressure through the windings of the yarn cake and intermittently halting the liquid flow and compressing the yarn cake as saponification proceeds and the back pressure exerted by the liquid saponifying medium drops.

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