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PREVENTION OF YELLOWING IN FRESHLY SPUN VISCOSE RAYON WITH TARTARIC, CITRIC OR GLUCONIC ACID

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This application is a continuation-in-part of copending application Serial No. 323,936, filed December 3, 1952, now abandoned.

This invention relates to the production of artificial fibers, and more particularly to a method for producing viscose rayon yarns.

One of the outstanding problems connected with the production of viscose rayon is the elimination of yellowing in the finished product. Various methods have been proposed to eliminate this discoloration, but no entirely satisfactory method is presently known for overcoming this disadvantage. This problem is particularly acute in the production of bleached viscose rayon yarn where it is unusually difficult to obtain the desired uniform whiteness throughout the yarn.

It is an object of the present invention to provide a process for the production of viscose rayon yarn having improved properties.

A further object of the invention is to provide a process for producing viscose rayon yarn characterized by the absence of yellowness.

A still further object of the invention is to provide a process for producing viscose rayon yarn with improved appearance, while at the same time making no sacrifices in the other physical properties of the yarn.

Other objects will be apparent hereinafter.

In accordance with the present invention, viscose yarn having improved optical properties is produced by aftertreating freshly spun yarn in the conventional manner with the exception that the liquid aftertreatment process concludes with a step of treating the yarn with a bath containing a non-volatile organic acid at a controlled pH. The yarn is then dried in the usual way.

The non-volatile acid treatment step of the present invention is preferably incorporated into that part of the aftertreating process when the yarn is treated with a yarn lubricant. A yarn lubricating bath which is also frequently referred to as a yarn finishing solution is conventionally the final step through which freshly spun yarn is aftertreated before the yarn is taken up on cones, warps, etc. For the preferred embodiment of the present invention any conventional yarn lubricant is satisfactory and the selection of a particular lubricant is by no means critical. A yarn lubricant gives the yarn a desired lubricity and pliability and may be in the form of an emulsion, solution or dispersion.

It is by no means essential to this invention that the non-volatile acid be incorporated into the yarn lubricating bath. The same inhibition of discoloration may be accomplished by subjecting the yarn to an aftertreating solution containing only the acid. But for convenience, the acid may be incorporated into the usual yarn lubricating bath thereby accomplishing the same effect without subjecting the yarn to an additional aftertreating step.

The invention has been found to be particularly useful in reducing the yellow color encountered in the manufacture of bleached cakes of viscose rayon, although it will be apparent that the invention is applicable to other

rayon aftertreating processes such as the so-called "continuous" method of producing viscose yarn. Very small quantities of a non-volatile organic acid in the final aftertreating bath have been found to be effective in eliminating any trace of discoloration in the finished product. For example, an aftertreating bath containing 0.045–0.055% tartaric acid is very effective in eliminating the slight yellow coloration encountered in the absence of the tartaric acid aftertreatment.

In a preferred embodiment of the invention, tartaric acid may be added to the final finishing bath in the production of viscose yarn. For example, a small amount of tartaric acid may be added to a yarn finishing bath containing an emulsion of water and mineral oil.

Yarn treated with a non-volatile organic acid in accordance with this invention may be dried in any conventional manner. When the treated yarn is subjected to a vacuum drying, a pronounced improvement in the color of the product is noted and the yellow coloration often present in vacuum dried yarn is avoided. If the acid treated yarn is dried by the so-called tunnel drying process, a definitely whiter yarn is obtained than in the case of ordinary tunnel dried yarn.

The improved color properties in the rayon yarn obtained by the non-volatile acid treatment is enhanced by careful control of the pH of the solution containing the acid. Particularly beneficial results are obtained if the pH of the acid containing finishing solution is maintained in the range of 3.3–3.7. At a pH of greater than about 4.2, the whitening effect of the acid treatment is considerably diminished. Any non-volatile organic acid capable of lowering the pH of the finishing solution within the above limits is useful in the present invention.

The amount of the non-volatile organic acid is not critical. However, the acid should be present in a sufficient amount to inhibit yellowness in the yarn but insufficient to be deleterious to the yarn.

The term "non-volatile" as used herein and in the appended claims is intended to mean a substance which does not vaporize or distill.

The invention will be further illustrated by the following examples:

EXAMPLE I

Sixteen cakes of 150/40 pot spun viscose rayon yarn, which had been aftertreated in the conventional manner including a treatment with soda bleach solution (containing 0.18–0.20% available chlorine as NaOCl) but which had not been treated with a finish, were pressure treated in the usual way at room temperature with a yarn lubricating bath containing 0.05% tartaric acid. The yarn lubricating bath exclusive of the added tartaric acid was an emulsion composed of about 98% water and 2% refined mineral oil. The pH of the finishing bath was 3.4. The cakes were then hydro-extracted in the usual way and placed in a vacuum drier at 20 mm. pressure and 78° C. for about 270 hours.

For purposes of comparison, several cakes of yarn from the same lot were treated with the same emulsion but with no tartaric acid added. These cakes were hydro-extracted in the usual manner and dried along with the acid treated cakes.

The dried cakes which had not been subjected to the tartaric acid treatment were found to possess a definite yellow color. The acid-treated cakes, on the other hand, appeared white to the eye. Color determinations on the two types of yarn indicated that the tartaric acid treated yarn was subsantially white in color while the untreated yarn was definitely yellow. The physical properties of the acid treated yarn were substantially equal to those for the regular yarn, and the acid treated samples did

not show any luster differences compared to the untreated samples.

EXAMPLE II

Sixteen cakes of 150/40 pot spun viscose rayon yarn which had been aftertreated as in Example I were pressure treated in the usual way at room temperature with a yarn finish emulsion of 98% water and 2% refined mineral oil to which 0.10% citric acid had been added. The pH of the finishing bath was about 3.5. Upon extracting and drying as in Example I, the yarn was compared with other cakes from the same lot which had been finished with the same finishing bath but containing no added acid. It was found that each of the acid finished cakes possessed a much whiter color than the cakes which had been finished in the normal manner. The physical properties of the acid treated yarn were substantially equal to those of the untreated yarn.

EXAMPLE III

Sixteen cakes of 150/40 pot spun viscose rayon yarn which had been aftertreated as in Example I were pressure treated in the usual manner at room temperature with a yarn finish emulsion of 98% water and 2% refined mineral oil to which 0.18% gluconic acid had been added. The pH of the finishing bath was about 3.8. After extracting and vacuum drying as in Example I, the acid treated yarn was found to be much whiter than yarn from the same lot which was treated with a finishing bath containing no added acid. The physical properties of the acid treated yarn were substantially unaffected by the acid treatment.

EXAMPLE IV

Large scale experiments were conducted in the production of cakes of 300/60 viscose rayon yarn by finishing the aftertreated cakes with an emulsion of 98% water and 2% refined mineral oil to which 0.045–0.55% tartaric acid had been added by pressure treatment in the usual way at room temperature. The pH of the finishing bath varied from 3.3 to 3.7. The treated cakes were extracted and dried as in the above examples. In all cases it was found that the yellow color normally accompanying the production of such yarn was entirely eliminated. The physical properties of the acid treated yarn were acceptable in all respects.

EXAMPLE V

Four cakes of 150/40 pot spun viscose rayon yarn which had been aftertreated as in Example I were pressure treated in the usual way at room temperature with a yarn finishing emulsion of 98½% water and 1½% refined mineral oil to which 0.06% tartaric acid had been added. The pH of the finishing bath was about 3.1. After extracting and vacuum drying as in Example I the acid treated yarn was found to be much whiter than yarn from the same lot which was treated with the same finishing bath containing no added acid. The physical properties of the acid treated yarn were substantially unaffected by the acid treatment.

EXAMPLE VI

Four cakes of 150/40 pot spun viscose rayon yarn which had been aftertreated as in Example I were pressure treated at room temperature with a final treatment with water containing 0.06% tartaric acid. The pH of the finishing solution was about 3.1. After extracting and vacuum drying as in Example I the acid treated yarn was found to be much whiter than yarn from the same lot which was treated with water containing no added acid. The physical properties of the acid treated yarn were substantially unaffected by the acid treatment.

In Table I below is listed data from color determination measurements made on samples of yarn treated in the above examples. Magnesium carbonate was used as a standard in these measurements. These color determination measurements were made on a Beckman model D.U. spectrophotometer as follows:

One reading, Y, of percent reflectance was taken at 580 millimicrons; another reading, B, of percent reflectance was taken at 460 millimicrons; the yellowness index was then calculated using the following formula:

$$\text{Index}=\frac{Y-B}{Y}\times 10^3$$

Table I

Sample		Color Determinations [1]	
		Outside Cake	Inside Cake
I	0.05% Tartaric Acid in Finish	0	12
II	0.10% Citric Acid in Finish	−7	−15
III	0.18% Gluconic Acid in Finish	−10	2
IV	0.05% Tartaric Acid in Finish	0	12
	Finish—No Acid	34	75
V	0.06% Tartaric Acid in Finish	−24	−32
	Finish—No Acid	−15	9
VI	0.06% Tartaric Acid in Water	−33	−34
	Water—No Acid	−14	0

[1] A color determination of 0 is considered white. A minus number is on the blue side; a plus number tends toward yellow.

The use of the non-volatile organic acids as aftertreating agents has several advantages. Many of them are common chemicals readily available in commercial quantities at moderate prices. They are readily incorporated into aftertreating baths and are non-injurious to processing equipment. Since they do not volatilize during the yarn drying process no corrosive fumes are produced. Tartaric acid, for example, has a very high melting point and a very low vapor pressure under processing conditions, and hence presents no corrosion problem which might be a factor if a more volatile material were used. Furthermore, these acids do not affect the stability of conventional finishing solutions in which they may be directly incorporated, if desired.

While the invention has been described with particular reference to a preferred method of carrying out its practice, other embodiments will be apparent to those skilled in the art. It is therefore intended to limit the invention only by the scope of the appended claims.

What is claimed is:

1. A method of aftertreating freshly spun viscose rayon yarn substantially free of impurities consisting of the steps of impregnating said yarn with an aqueous solution consisting essentially of a small amount of a non-volatile aliphatic organic acid selected from the group consisting of tartaric acid, citric acid and gluconic acid, said solution having a pH produced by said acid of less than about 4.2 and insufficient to appreciably affect the physical properties of the yarn, and thereafter drying for the first time the acid impregnated yarn, the resulting yarn being characterized by the absence of yellow coloration.

2. The method of claim 1 when the non-volatile acid is tartaric acid.

3. The method of claim 1 when the non-volatile acid is gluconic acid.

4. The method of claim 1 when the non-volatile acid is citric acid.

5. A method of aftertreating a freshly spun cake of viscose rayon yarn substantially free of impurities consisting of impregnating said cake with an aqueous solution consisting essentially of a non-volatile aliphatic organic acid selected from the group consisting of tartaric acid, citric acid and gluconic acid, said solution having a pH produced by said acid of less than about 4.2 and insufficient to appreciably affect the physical properties of the yarn, and thereafter drying for the first time the acid impregnated viscose yarn under vacuum, the

resulting yarn being characterized by the absence of yellow coloration.

6. A method for the aftertreating of freshly spun viscose rayon yarn substantially free of impurities consisting of impregnating said yarn with a yarn finishing bath consisting essentially of an aqueous medium containing an inert textile lubricant and a non-volatile aliphatic organic acid selected from the group consisting of tartaric acid, citric acid and pluconic acid, said finishing bath having a pH produced by said acid of less than about 4.2 and insufficient to appreciably affect the physical properties of the yarn, and thereafter drying for the first time the acid impregnated yarn.

7. The method of claim 6 wherein the drying of the acid impregnated yarn is accomplished under vacuum.

8. The method of claim 6 wherein tartaric acid is incorporated into the yarn finishing bath.

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