

1

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## TREATING FILAMENTARY MATERIAL

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This invention relates to the wet spinning of cellulose triacetate to form filamentary materials.

Textile materials of cellulose triacetate have recently attained considerable commercial importance. On suitable after-treatment, these textile materials have superior heat resistance, high safe-ironing temperatures, excellent wash-fastness even when dyed in heavy shades, crease resistance and resistance to glazing and to shrinkage on pressing with moist steam.

It has recently been found that cellulose triacetate filamentary materials of greater strength and high elongation can be prepared by wet spinning of the cellulose triacetate, i.e. by extrusion of a solution of the cellulose triacetate into a coagulating or "spin" bath which exerts a swelling action on the filamentary material. Upon contacting a plurality of such filaments while wet and swollen with the spin bath and then drying, it has been found that the filaments tend to coalesce to an appreciable extent, i.e. they bond laterally to one another.

It is accordingly an object of this invention to provide a process for reducing the coalescence of wet spun cellulose triacetate filamentary material.

It is another object of the invention to provide a continuous process for wet spinning cellulose triacetate and recovering filaments thereof in substantially uncoalesced condition.

Other objects of this invention will be apparent from the following detailed description and claims. In this description and claims all proportions are by weight unless otherwise indicated.

In accordance with one aspect of this invention, a solution of cellulose triacetate in a solvent comprising methylene chloride is extruded through a spinnerette, preferably provided with a plurality of orifices, into a spin bath containing methylene chloride and preferably a lower aliphatic alcohol such as methanol. The resulting swollen cellulose triacetate filaments are then treated with a liquid organic non-solvent for cellulose triacetate. It is found that upon drying the filaments are uncoalesced.

The formation of the swollen cellulose triacetate filaments is preferably carried out in the manner described in detail in the copending application of Jesse L. Riley, entitled Spinning, Serial No. 638,414, filed February 5, 1957, and the entire disclosure of said Riley application is hereby incorporated into the present application.

As taught in the copending application of Riley, referred to above, the use of certain critical proportions of methylene chloride in the spin bath results in the production of filamentary materials of outstanding physical properties (e.g. tenacity of at least 1.8 grams per denier and elongation of at least 18%). In the practice of the present invention it is preferable to use proportions of methylene chloride within the scope of the Riley application. Preferably the proportions of methylene chloride in the spin bath are within the range defined by the formula  $C=75\frac{1}{2}-T\pm 5$ , where C is the concentration in percent and T is the temperature in degrees centigrade, the balance of the solvent comprising a lower alkanol, preferably methanol. Since the temperature of the spin bath preferably ranges from about 15 to 45° C., the concentration of methylene chloride will range between about 25 and 65% although preferably it is less than 50%. As disclosed in said Riley application, the resulting filaments are stretched in the spin-bath, which

2

has a considerable swelling power for the cellulose triacetate, the stretching being effected by taking up the swollen filaments at a higher linear speed than the linear speed at which they are extruded. The stretching force is most conveniently applied by means of a positively driven feed roll, such as a godet roll, over which the filaments pass, as a bundle, after they leave the spin-bath.

The liquid organic non-solvent for cellulose triacetate which can be employed to treat the bundle of cellulose triacetate filaments from the spin bath is preferably miscible with methylene chloride and boils below 150° C. Representative substances include hydrocarbons such as aliphatic compounds and mixtures thereof, cycloaliphatic compounds such as cyclohexane, aromatic compounds such as benzene and its lower alkyl substitution products such as toluene and xylene, ethers such as isopropyl ether, and the like. The use of xylene is particularly satisfactory since it permits a marked reduction in coalescence in a short time of contact, and the filaments have desirable physical properties such as high strength and satisfactory elongation. The bundle of filaments can be immersed freely in the treating bath after leaving the spin-bath without being taken up on a bobbin. Preferably, however, the bundle of filaments leaving the spin-bath is passed about a positively driven feed roller, through a second bath containing the treating liquid and is taken up on a bobbin. Guides can be provided in the second bath to maintain the yarn submerged therein. It is generally advantageous to maintain a low tension on the yarn during the treatment of this invention, so as to produce a final product of desirable high elongation with a low degree of coalescence. To this end the take-up bobbin may be run at a somewhat lower linear velocity than the feed roller.

With certain of the treating liquids, such as cyclohexane, the percent coalescence can be reduced by mechanically assisting the treatment, as by the use of suitable wiping devices in the treating bath, e.g. an aluminum magnesium silicate device having a satin-finished rounded wiping edge which will flatten the filament bundle without appreciably increasing the tension on the filaments.

The temperature of the treating bath may range from room temperature up to the boiling point of the spin-bath components, e.g. up to about 60° C. Temperatures ranging from about 20° C. to 40° C. are preferred.

Drying can be effected in any manner, such as by standing, by heating, by vacuum, by an air jet, or the like. It can conveniently be effected on the bobbin or it can be effected with the filaments relaxed, i.e. filamentary material merely passes about a roller and is collected in a can where it is free to contract.

The cellulose triacetate employed in accordance with this invention has an acetyl value of at least about 60%, preferably above 61%, calculated as acetic acid. Its concentration in the spinning solution can range between about 18 to 26% by weight. The spinning solution solvent can contain up to about 15% by weight of methanol while the methanol content of the spin-bath is about 50% by weight.

The following examples are given to illustrate this invention further. The percent coalescence as employed in the examples is that percentage of the total number of filaments in a yarn bundle which participate in coalescence. It is found as follows: A piece of piano wire of 0.097 mm. diameter is looped through a hole of 1.02 mm. diameter in a metal plate 0.62 mm. thick. A 10 cm. length of uncolored yarn to be examined is banded with a blue yarn and the bundle is inserted through the loop of the wire. The wire loop is pulled through the hole in the plate, thereby doubling the filament bundle and draw-

ing it through the hole. The blue yarn serves to fill the hole and ensures a snug fit. The yarn is cut flush with the surfaces of the plate using a razor blade, thereby forming a thin section. A drop of decane is placed on the section to improve filament boundary definition, a cover glass is placed over the section and it is examined under a microscope at a magnification of 440. The number of filaments which are fused or bonded together is determined and the test repeated 8 times for each yarn. The percent coalescence is 100 times the average number of filaments of a section involved in coalescence divided by the total number of uncolored filaments in the section.

#### EXAMPLE I

Cellulose triacetate of 61.5% acetyl value, calculated as acetic acid, and of intrinsic viscosity 2.0 (measured in cupriethylene diamine at 25° C. on cellulose regenerated from said cellulose triacetate) is dissolved in a solvent comprising 91 parts by weight of methylene chloride and 9 parts by weight of methanol to form a 22% by weight solution having a viscosity of 1800 poises at 25° C. The solution is spun upwardly through a spinning jet having 40 holes each 0.10 mm. in diameter, the filaments passing upwardly through 1.7 meters of a spin-bath composed by weight of 42.2/57.8 methylene chloride/methanol. The spin-bath is maintained at 35° C. and is circulated upwardly at the rate of 4.0 liters/minute through a vertical column of 2 inches internal diameter. After emerging from the bath, the yarn is passed over a ball-bearing pulley and 3 meters downward to a driven feed roll and idler operating at a surface speed of 60 meters/minute. From the feed roll, the yarn passes through a 1.7 meter horizontal bath of xylene at room temperature to a take-up bobbin rotated at a peripheral speed of 57-60 meters/minute. The yarn is dried on the bobbin by standing in air for 17 hours, then standing for 17 hours at 50° C. and an absolute pressure of 200 mm. of mercury, and finally standing for 20 hours at 50° C. and an absolute pressure of 5 mm. of mercury. The percent coalescence of the yarn is 5.0%.

#### EXAMPLE II

Solutions of cellulose triacetate as described in Example I are spun vertically upwardly through a 1.0 meter height of spinning bath made up of a mixture of methylene chloride and methanol. The filaments pass about a godet and idler assembly, through a horizontal treating bath 95 cm. long, and then to a take-up roll, fixed guides being employed to submerge the yarn in the bath. The results using a variety of conditions and solvents are given in the following table:

Table

Yarn: 40 filaments.  
Spinning Speed: 40 m./min. (3.75 denier); 23.5 m./min. (6.38 denier).  
Spin Bath: 1 meter CH<sub>2</sub>Cl<sub>2</sub>/CH<sub>3</sub>OH.  
Treating Bath: 95 cm.

| Treating Liquid      | Spinning Speed, m./min. | CH <sub>2</sub> Cl <sub>2</sub> Content of CH <sub>2</sub> Cl <sub>2</sub> /CH <sub>3</sub> OH Spin bath, Wt. percent | Treating Temp., ° C. | Bath Immersion Time, sec. | Takeup Tension, gm. | Coalescence, percent |
|----------------------|-------------------------|-----------------------------------------------------------------------------------------------------------------------|----------------------|---------------------------|---------------------|----------------------|
| None.....            | 40                      | 50                                                                                                                    | -----                | -----                     | 5                   | 50.                  |
|                      | 40                      | 41                                                                                                                    | -----                | -----                     | 10                  | 25.0.                |
| Xylene.....          | 40                      | 41                                                                                                                    | 35                   | 1.4                       | 7                   | 2.5.                 |
|                      | 40                      | 41                                                                                                                    | 35                   | 1.4                       | 3                   | 0.                   |
| Benzene.....         | 40                      | 50                                                                                                                    | 25                   | 1.4                       | 10                  | 10.0.                |
|                      | 40                      | 50                                                                                                                    | 25                   | 17 hours <sup>1</sup>     | 0                   | 16.2.                |
| Isopropyl Ether..... | 40                      | 50                                                                                                                    | 20                   | 1.4                       | 7-10                | 13.                  |
|                      | 40                      | 50                                                                                                                    | 20                   | 1.4                       | 5                   | 6.3.                 |
|                      | 40                      | 50                                                                                                                    | 25                   | 1.4                       | 9                   | 11.2.                |
| Cyclohexane.....     | 40                      | 50                                                                                                                    | 25                   | 1.4+300 <sup>2</sup>      | 10                  | 5.0.                 |
|                      | 40                      | 50                                                                                                                    | 15                   | 30 <sup>1</sup>           | 0                   | 2.5.                 |
|                      | 23.5                    | 41                                                                                                                    | 35                   | 2.5                       | 6                   | 12.5.                |
| Water.....           | 40                      | 50                                                                                                                    | 25                   | 1.4                       | ~3                  | Almost 100.          |

<sup>1</sup> Yarn taken up on bobbin immediately after spinning, removed from bobbin and immersed freely.

<sup>2</sup> After travelling filaments treated in bath and taken up on bobbin, bobbin immersed in treatment bath.

When water is used in the treating bath not only are the filaments almost fully coalesced as indicated in the table, but the yarn is also exceedingly weak and shatters rather than breaking cleanly.

It is to be understood that the foregoing detailed description is merely given 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 to secure by Letters Patent is:

1. Process for the production of filamentary cellulose triacetate which comprises extruding a solution of cellulose triacetate in a solvent therefor comprising methylene chloride into a spin bath comprising about 25 to 65% of methylene chloride and a lower aliphatic alcohol to form filamentary cellulose triacetate, and treating said filamentary cellulose triacetate with a liquid organic non-solvent for cellulose triacetate which is miscible with methylene chloride and boils below about 150° C.

2. The process set forth in claim 1, wherein the filamentary cellulose triacetate upon leaving said spin bath passes continuously through a treating bath containing said liquid organic non-solvent for cellulose triacetate.

3. The process set forth in claim 1, wherein the solvent from which the cellulose triacetate is spun comprises methylene chloride and up to about 15% by weight of a lower alkanol, and the treatment with said liquid organic non-solvent for cellulose triacetate is effected with the filamentary cellulose triacetate under low tension.

4. The process set forth in claim 1, wherein the spin bath comprises a mixture of methylene chloride and methanol containing at least about 50% by weight of methanol.

5. The process set forth in claim 1, wherein the filamentary cellulose acetate upon leaving said spin bath is taken up on a bobbin, and said bobbin is thereafter immersed in a treating bath containing said liquid organic non-solvent for cellulose triacetate.

6. The process set forth in claim 1, wherein said liquid organic non-solvent for cellulose triacetate comprises isopropyl ether.

7. The process set forth in claim 1, wherein said liquid organic non-solvent for cellulose triacetate comprises a cyclic hydrocarbon.

8. The process set forth in claim 1, wherein said liquid organic non-solvent for cellulose triacetate comprises cyclohexane.

9. The process set forth in claim 1, wherein said liquid organic non-solvent for cellulose triacetate comprises a monocyclic aromatic hydrocarbon.

2,999,004

5

10. The process set forth in claim 1, wherein said liquid organic non-solvent for cellulose triacetate comprises xylene.

11. The process set forth in claim 1, wherein said liquid organic non-solvent for cellulose triacetate comprises benzene. 5

12. The process set forth in claim 1, wherein treating is effected by continuously introducing said filamentary cellulose triacetate into a treating bath and removing it from said treating bath at a lower linear velocity than it is introduced, treating thereby being effected with said filamentary cellulose triacetate under low tension. 10

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