

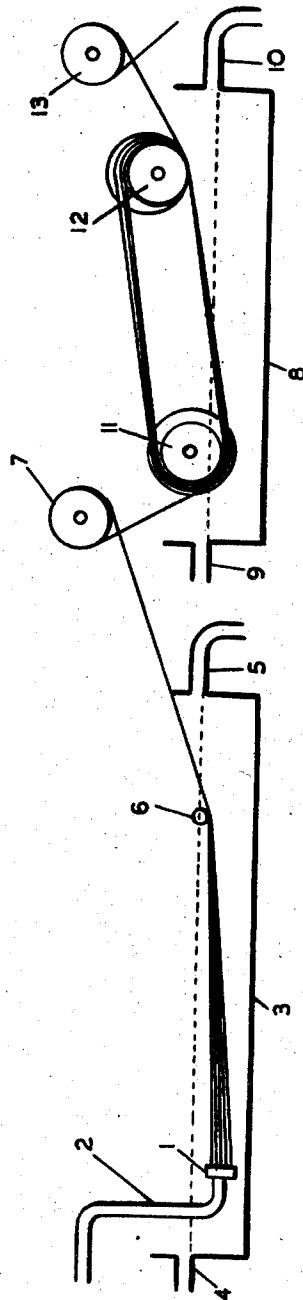
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PRODUCTION OF CELLULOSE ESTER TEXTILE MATERIALS

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PRODUCTION OF CELLULOSE ESTER
TEXTILE MATERIALS

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This invention relates to the manufacture of cellulose ester yarns suitable for conversion into staple fibre.

In one method of making staple fibre from cellulose esters, in particular cellulose acetate, a tow or untwisted bundle of filaments is made and cut up; the tow is made up from untwisted yarns of high denier, e. g. about 500-5,000 or more, which in turn are preferably made up from filaments of denier about 1.5-3.0; the yarns should be moderately strong and of good extensibility; for example they preferably have a tenacity of about 2.0-2.5 grammes per denier or more and a minimum extensibility of about 15%.

A process to be suitable for making cellulose acetate material for conversion into staple fibre by this method should therefore be capable of producing yarns of high denier having the properties outlined above; furthermore it should be capable of operation at fairly high spinning speeds, and should make use of solvents and other agents that are easily made and recovered. The present invention provides a means of satisfying these requirements.

According to the invention, yarns having a basis of a lower aliphatic ester of cellulose and suitable for conversion into staple fibre are made by a process which comprises forming filaments from the cellulose ester by extruding an acetone solution of the cellulose ester into an aqueous solution of diethylene glycol diacetate of concentration below 38% as coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments to the action of a regulating bath comprising an aqueous solution of diethylene glycol diacetate of concentration below 38% while allowing them to shrink freely. The filaments may then be washed substantially free from the diethylene glycol diacetate, for instance by means of cold or warm water, and dried. The invention is of particular importance for the manufacture of yarns from cellulose acetate, and it will be described with particular reference to this material.

It is very advantageous to subject the filaments in the form of untwisted yarn to the action of the regulating bath while passing over advancing rollers or a single advancing reel so shaped or adjusted as to allow the yarn to shrink freely.

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Generally it is sufficient to allow for a shrinkage of about 2%-4%, and this may be done by using a pair of slightly tapered rollers, one of which is fixed skew to the other so that the yarn travels across the rollers in the direction of the taper. Preferably one of the rollers dips into the regulating bath, but other methods of applying the bath to the yarn may be employed. For example the regulating bath may be sprayed onto or otherwise applied to the yarn during its passage through the atmosphere over or between the rollers, or it may be applied by means of hollow perforated rollers. (It will be understood that, while, for convenience, the term "regulating bath" is employed to denote the diethylene glycol diacetate solution with which the yarn is treated as described above, the word "bath" is not intended to imply the use of any particular method of applying the solution to the yarn.)

It is preferred to employ as the regulating bath a diethylene glycol diacetate solution of the same concentration as that used in the coagulating bath, but at a lower temperature. Advantageously the coagulating bath may comprise a diethylene glycol diacetate solution of concentration about 30%-35% kept at a temperature between about 30° and 35° C. The temperature of the regulating bath is preferably about 8°-15° C. lower, i. e. between about 15° and 27° C. according to the temperature of the coagulating bath. If desired however the temperature of the regulating bath may be approximately the same as that of the coagulating bath and/or its concentration may be lower.

The spinning jet and coagulating bath may be arranged for either horizontal or vertical, preferably upward, spinning. The best length of the coagulating bath and length of travel of the yarn in contact with the regulating bath depend on a number of factors, including filament denier, the number of filaments in the yarn, the rate of travel of the yarn and the rate at which spent liquor in the coagulating and regulating baths is replaced by fresh. It is in any case very desirable that the yarn should be substantially free from acetone before it leaves the regulating bath. As a general rule it may be said that the path of the yarn through the coagulating bath may be between about 50 and 90 cm. and the path of the

yarn in contact with the regulating bath between about 3 and 10 m.

After its treatment with the regulating bath, the yarn may be given a thorough wash with water to remove substantially all the diethylene glycol diacetate which it contains. For example it may be passed through one or more vessels containing water, or in counter-current to a stream of water flowing in a trough or the like. Preferably, however, at least part of the washing is effected by soaking the yarn in water a number of times and then running off the water or removing the yarn from the water, or by causing water to flow continuously over the yarn for some time, e. g. about 1-4 hours; advantageously a continuous wash of this kind may be followed by a wash in which the yarn is allowed to stand in water for several hours. The diethylene glycol diacetate can be recovered from the wash water, for example by extraction with benzene, and used again in a coagulating or regulating bath. If desired, the wash water may be partly or wholly replaced by an organic solvent for diethylene glycol diacetate which is without action on cellulose acetate, e. g. petroleum ether.

A suitable form of device for carrying out the invention is illustrated diagrammatically in the drawing.

The device comprises a spinning jet 1 containing, for example, 200-1,000 holes of diameter 0.08-0.12 mm., fed by a pipe 2 from a spinning pump and filters (not shown) and situated below the liquid level in a horizontal trough 3 provided with a liquid inlet 4 and overflow 5. The trough is kept filled to the overflow level with a 30%-35% aqueous diethylene glycol diacetate solution at a temperature kept between 31° and 33° C., which enters continuously through the inlet 4 and runs off by the overflow 5. Towards the further end of the trough and about at the level of the surface of the liquid is a guide rod 6 adapted to lead the filaments formed in the coagulating bath as a single untwisted yarn to a godet 7.

In line with the trough 3 is situated a second horizontal trough B provided with a liquid inlet 9 and overflow 10 for the regulating bath, and positioned above it a pair of tapered advancing rollers 11 and 12, one of which dips below the surface of the liquid in the trough 8. The regulating bath is a solution of diethylene glycol diacetate of the same concentration as that of the coagulating bath but kept at a temperature between 21° and 23° C. The rollers 11 and 12 both have a 1% taper along their length and one of them is mounted in adjustable bearings so that its axis can be adjusted to make any desired angle with the axis of the other and so cause the yarn to travel down the rollers in the direction of the taper. A second godet 13 is situated so as to receive the yarn leaving the advancing rollers. The godet 7 is preferably driven at such a speed as to impart to the filaments in the coagulating bath as high a degree of stretch as they will take without breaking, and godet 13 at a suitably lower speed to allow the yarn passing between the two godets and round the advancing rollers to shrink freely, e. g. at a peripheral speed 4% less than that of the godet 7.

In a modification of the invention, the regulating bath may comprise water alone instead of aqueous diethylene glycol diacetate, and in this case also the temperature is preferably, though not necessarily, below that of the coagulating bath. When using water the yarn does not as a rule shrink to any substantial degree, and it is

therefore advisable to employ cylindrical rather than tapered advancing rollers so as to avoid sagging without subjecting the yarn to any substantial degree of tension. However, since the regulating bath of aqueous diethylene glycol diacetate gives a product of greater tenacity and extensibility, its use is preferred.

Cellulose acetate yarns produced in accordance with the invention not only possess good tenacity and extensibility but also frequently have a considerable degree of crimp, making them very suitable for conversion into staple fibre.

Although the regulating bath is preferably of the same composition and may also be at the same temperature as the coagulating bath, its action differs from that of the coagulating bath, and the effect of employing a separate regulating bath is quite different from the effect of correspondingly increasing the time of travel of the yarn through the coagulating bath.

While the invention has been described with particular reference to cellulose acetate, it is also applicable to the production of yarn from other lower aliphatic acid esters of cellulose, especially cellulose propionate and acetate propionate.

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

1. The process for the manufacture of yarns having a basis of a lower aliphatic ester of cellulose and suitable for conversion into staple fibre, which comprises forming filaments from the cellulose ester by extruding through a multi-hole spinning jet an acetone solution of the cellulose ester into an aqueous solution of diethylene glycol diacetate of concentration below 38% as coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone, to the action of a regulating bath comprising an aqueous solution of diethylene glycol diacetate of concentration below 38% while allowing them to shrink freely.

2. Process for the manufacture of yarns having a basis of cellulose acetate and suitable for conversion into staple fibre, which comprises forming filaments from the cellulose acetate by extruding through a multi-hole spinning jet an acetone solution of the cellulose acetate into an aqueous solution of diethylene glycol diacetate of concentration below 38% as coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone, to the action of a regulating bath comprising an aqueous solution of diethylene glycol diacetate of concentration below 38% while allowing them to shrink freely.

3. Process for the manufacture of yarns having a basis of a lower aliphatic ester of cellulose and suitable for conversion into staple fibre, which comprises forming filaments from the cellulose ester by extruding through a multi-hole spinning jet an acetone solution of the cellulose ester into an aqueous solution of diethylene glycol diacetate of concentration 30% to 35% as coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone, to the action of a regulating bath comprising an aqueous solution of diethylene glycol diacetate of concentration 30% to 35% while allowing them to shrink freely.

4. Process for the manufacture of yarns having a basis of cellulose acetate and suitable for

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conversion into staple fibre, which comprises forming filaments from the cellulose acetate by extruding through a multi-hole spinning jet an acetone solution of the cellulose acetate into an aqueous solution of diethylene glycol diacetate of concentration 30% to 35% as coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone, to the action of a regulating bath comprising an aqueous solution of diethylene glycol diacetate of concentration 30% to 35% while allowing them to shrink freely.

5. Process for the manufacture of yarns having a basis of a lower aliphatic ester of cellulose and suitable for conversion into staple fibre, which comprises forming filaments from the cellulose ester by extruding through a multi-hole spinning jet an acetone solution of the cellulose ester into an aqueous solution of diethylene glycol diacetate of concentration 30% to 35% and at a temperature of 30° to 35° C. as coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone, to the action of a regulating bath consisting of an aqueous solution of diethylene glycol diacetate of the same concentration as the coagulating bath, and at a temperature 8° to 15° C. lower, while allowing them to shrink freely.

6. Process for the manufacture of yarns having a basis of cellulose acetate and suitable for conversion into staple fibre, which comprises forming filaments from the cellulose acetate by extruding through a multi-hole spinning jet an acetone solution of the cellulose acetate into an aqueous solution of diethylene glycol diacetate of concentration 30% to 35% and at a temperature of 30° to 35° C. as coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone, to the action of a regulating bath consisting of an aqueous solution of diethylene glycol diacetate of the same concentration as the coagulating bath, and at a temperature 8° to 15° C. lower, while allowing them to shrink freely.

7. Process for the manufacture of yarns having a basis of a lower aliphatic ester of cellulose and suitable for conversion into staple fibre, which comprises forming filaments from the cellulose ester by extruding through a multi-hole spinning jet an acetone solution of the cellulose ester into an aqueous solution of diethylene glycol diacetate of concentration below 33% as coagulating bath, collecting the filaments into an untwisted yarn and applying to the yarn a tension such that the filaments are stretched during their passage through the coagulating bath, and then subjecting the yarn, until it is substantially free from acetone, to the action of a regulating bath consisting of an aqueous solution of diethylene glycol diacetate of concentration below 38%, while allowing it to shrink freely.

8. Process for the manufacture of yarns having a basis of cellulose acetate and suitable for conversion into staple fibre, which comprises forming filaments from the cellulose acetate by extruding through a multi-hole spinning jet an acetone solution of the cellulose acetate into an aqueous solution of diethylene glycol diacetate of concentration 30% to 35% as coagulating bath, collecting the filaments into an untwisted yarn

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and applying to the yarn a tension such that the filaments are stretched during their passage through the coagulating bath, and then subjecting the yarn, until it is substantially free from acetone, to the action of a regulating bath consisting of an aqueous solution of diethylene glycol diacetate of concentration 30% to 35%, while allowing it to shrink freely.

9. Process for the manufacture of yarns having a basis of cellulose acetate and suitable for conversion into staple fibre, which comprises forming filaments from the cellulose acetate by extruding through a multi-hole spinning jet an acetone solution of the cellulose acetate into an aqueous solution of diethylene glycol diacetate of concentration 30% to 35% as coagulating bath, collecting the filaments into an untwisted yarn and applying to the yarn a tension such that the filaments are stretched during their passage through the coagulating bath, and then subjecting the yarn, until it is substantially free from acetone, to the action of a regulating bath consisting of an aqueous solution of diethylene glycol diacetate of concentration 30% to 35%, while allowing it to shrink freely, the number and dimensions of the holes in the spinning jet and the degree of stretch being so chosen that the yarn, after it has shrunk, has a denier above 500 while the individual filaments have a denier below 3.

10. Process for the manufacture of yarns having a basis of cellulose acetate and suitable for conversion into staple fibre, which comprises forming filaments from the cellulose acetate by extruding through a multi-hole spinning jet an acetone solution of the cellulose acetate into an aqueous solution of diethylene glycol diacetate of concentration 30% to 35% at a temperature of 30° to 35° C. as coagulating bath, collecting the filaments into an untwisted yarn and applying to the yarn a tension such that the filaments during their passage through the coagulating bath are stretched to substantially the highest degree possible without breaking them, and then subjecting the yarn, until it is substantially free from acetone, to the action of a regulating bath consisting of an aqueous solution of diethylene glycol diacetate of the same concentration as the coagulating bath and at a temperature 8° to 15° C. lower while allowing it to shrink freely.

11. Process for the manufacture of yarns having a basis of cellulose acetate and suitable for conversion into staple fibre, which comprises forming filaments from the cellulose acetate by extruding through a multi-hole spinning jet an acetone solution of the cellulose acetate into an aqueous solution of diethylene glycol diacetate of concentration 30% to 35% at a temperature of 30° to 35° C. as coagulating bath, collecting the filaments into an untwisted yarn and applying to the yarn a tension such that the filaments during their passage through the coagulating bath are stretched to substantially the highest degree possible without breaking them, and then subjecting the yarn, until it is substantially free from acetone, to the action of a regulating bath consisting of an aqueous solution of diethylene glycol diacetate of the same concentration as the coagulating bath and at a temperature 8° to 15° C. lower while allowing it to shrink freely, the number and dimensions of the holes in the spinning jet being so chosen that the yarn, after it has shrunk, has a denier above 500 while the individual filaments have a denier below 3.

12. Process according to claim 1, wherein the

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yarn is subjected to the action of the regulating bath while on a thread-advancing thread-storage device.

13. Process according to claim 8, wherein the yarn is subjected to the action of the regulating bath while on a thread-advancing thread-storage device. 5

14. Process according to claim 11, wherein the yarn is subjected to the action of the regulating bath while on a thread-advancing thread-storage device. 10

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