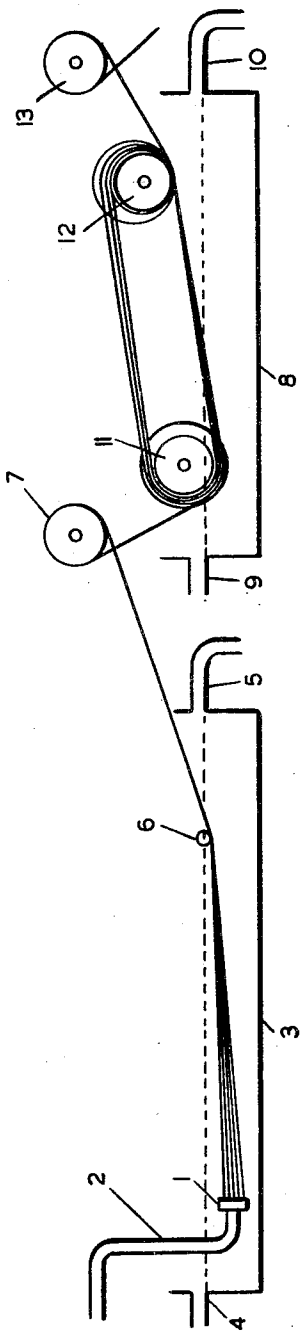
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PRODUCTION OF CELLULOSE ESTER YARNS SUITABLE FOR CONVERSION INTO STAPLE FIBER

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PRODUCTION OF CELLULOSE ESTER YARNS SUITABLE FOR CONVERSION INTO STAPLE FIBER

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This invention relates to the production of cellulose ester yarns suitable for conversion into staple fibres.

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 to 5000 or more, which in turn are preferably made from filaments of denier about 1.5 to 3.0; the yarns should be moderately strong and of good extensibility; for example they preferably have a tenacity of about 2.0 to 2.5 grammes per denier or more and a minimum extensibility of about 15%.

A process to be suitable for making cellulose ester 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.

In our co-pending application S. No. 109,274 filed August 9, 1949 we have described a process which satisfies these requirements, wherein filaments are formed from a cellulose ester of an aliphatic acid containing up to 4 carbon atoms, especially cellulose acetate, by extruding an acetone solution of the cellulose ester through a multi-hole spinning jet into an aqueous solution of diethylene glycol diacetate of concentration below 38%, and especially 30–35%, 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 below 38%, and especially 30–35%, while allowing them to shrink freely. The filaments may then be washed substantially free from diethylene glycol diacetate, for instance by means of cold or warm water, and dried.

We have now found that in place of the diethylene glycol diacetate in the coagulating or regulating bath or both we may employ a mixture of diethylene glycol diacetate and diethylene glycol monoacetate, provided the total amount of combined acetic acid corresponds to that in an aqueous diethylene glycol diacetate solution of concentration below 38%, and especially between 30% and 35%. Preferably the mixture of diethylene glycol acetates consists of 88% or more of the diacetate and 12% or less of

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the monoacetate, but mixtures containing lower proportions of the diacetate, for instance down to 80% or even lower, can be employed if desired.

It is very advantageous to subject the filaments in the form of untwisted yarn to the action of the regulating bath while the yarn is passing over advancing rollers or over a single advancing reel, that is to say, a set or pair of rollers or a single reel adapted to carry a number of convolutions of the yarn and in rotating to cause the convolutions to travel successively along the rollers or reel in an axial direction. The rollers or reel should be so shaped or adjusted as to allow the yarn to shrink freely. Generally it is sufficient to allow for a shrinkage of about 2% to 4%, and this may for example 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 acetate solution with which the yarn is treated as described above, the word "bath" is not intended to denote any particular method of applying the solution to the yarn.)

It is preferred to employ as the regulating bath a diethylene glycol acetate solution of the same concentration of combined acetic acid as that used in the coagulating bath, but at a lower temperature. Advantageously, a coagulating bath having a combined acetic acid concentration equal to that of a diethylene glycol diacetate solution of concentration about 30% to 35% is kept at a temperature of about 30° to 35° C. The temperature of the regulating bath is preferably about 8° to 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 its concentration in terms of combined acetic acid 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 regulating and coagulating 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 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 acetate which it contains; for example it may be passed through one or more vessels containing water or in countercurrent to a stream 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 running off the water or removing the yarn from the water, or by causing water to flow continuously over the yarn for some considerable time, e. g. about 1 to 4 hours; advantageously, a wash by one or more of these methods may be followed by a wash in which the yarn is allowed to stand in water for several hours. The ethylene glycol acetates can be removed from the wash water, for example by extraction with benzene, and used again in a regulating or coagulating bath. If desired, the wash water may be partly or wholly replaced by an organic solvent for the diethylene glycol acetates which is substantially without action on the cellulose ester, 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 acetate 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 8 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 acetates 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 or 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.

An important application of the discovery underlying the present invention lies in the repeated use, as coagulating or regulating bath or both, of a solution which may initially consist of water and diethylene glycol diacetate only. It is our experience that in course of time part of the diethylene glycol diacetate in such a solution is hydrolysed to diethylene glycol monoacetate and free acetic acid. The resulting change in composition of the solution will, unless dealt with, cause a change in the properties of the yarn obtained and may also give rise to spinning difficulties.

As a result of our discovery, we are now able to keep substantially constant the effect exerted by the solution (whether used as a coagulating or as a regulating bath) even though its composition alters, by adding to the solution sufficient fresh diethylene glycol diacetate, diethylene glycol monoacetate, or both, to keep its content of combined acetic acid constant. Thus, for example, if 2% of the diethylene glycol diacetate originally present has undergone hydrolysis to the monoacetate, the solution may be restored to its original effectiveness by adding a further 1% only of diethylene glycol diacetate, or so much of a mixture of the diacetate and monoacetate (such for instance as may be recovered from the wash water as already described) as will have the same content of combined acetic acid.

Before re-using the solution, it is advisable to ensure that its acetone content (derived from the spinning solution) does not exceed 1%. Thus acetone may be removed from the solution by evaporation, for example in an evaporator of the climbing film type. It is also advisable to keep the content of free acetic acid (derived from the diethylene glycol diacetate by its hydrolysis) to a maximum of 1% and preferably below about 0.5%. For example, when the acetic acid concentration has reached the desired upper limit, the solution may be heated, preferably under reduced pressure, so as to remove acetic acid by distillation in the form of an azeotropic mixture with water. If desired both acetic acid and acetone may be removed in a single operation. Before the solution from which the acetic acid has been removed is used again, it may be necessary to replace part or all of the water removed with the acetic acid, so as to keep the concentration of combined acetic acid constant.

It is possible instead of removing acetic acid to neutralise it, for example by means of an alkali or alkaline earth metal carbonate or hydroxide, but this is usually less desirable since the neutralisation products tend to accelerate further hydrolysis of the diethylene glycol diacetate.

In a modification of the invention, the regulating bath may comprise water alone instead of an aqueous diethylene glycol acetate solution, 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 advancing rollers or an advancing reel which does not allow for shrinkage, e. g. a pair of cylindrical rather than tapered rollers, so that sagging is avoided without subjecting the yarn to any substantial degree of tension. Usually a product of greater tenacity and extensibility is obtained when the regulating bath consists of an aqueous diethylene

glycol acetate solution, but in particularly hot weather a regulating bath of water alone may sometimes be preferable to a solution of diethylene glycol diacetate with or without a proportion of the monoacetate.

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

While the process of the invention is particularly suitable for making cellulose acetate yarn for cutting up into staple fibres, it is also applicable to the production of yarn from other cellulose esters of aliphatic acids containing up to 4 carbon atoms, especially cellulose propionate and acetate propionate.

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

1. 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 an acetone solution of the cellulose ester through a multi-hole spinning jet into a coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone and while allowing them to shrink freely, to the action of a regulating bath, the coagulating and regulating baths both being aqueous solutions selected from the group which consists of aqueous solutions of diethylene glycol diacetate and aqueous solutions of mixtures of diethylene glycol diacetate and monoacetate in which the amount of diacetate by weight is at least 4 times that of monoacetate, the concentration of the solutions being such that their content of combined acetic acid is equal to that of a diethylene glycol diacetate solution of concentration below 38%, at least one of the baths containing both the diacetate and the monoacetate, removing the coagulating and regulating baths from their respective zones of operation, removing from said baths by distillation substantially all of the acetone derived from the spinning solutions, adding to the baths sufficient diethylene glycol diacetate to restore their initial concentration of combined acetic acid while maintaining the amount of diethylene glycol diacetate in each bath at at least 88% of the total weight of diethylene glycol diacetate and monoacetate in the bath, and returning the baths to their respective zones of operation.

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 an acetone solution of the cellulose acetate through a multi-hole spinning jet into a coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone and while allowing them to shrink freely, to the action of a regulating bath, the regulating and coagulating baths both being aqueous solutions of a mixture of diethylene glycol diacetate and monoacetate in which the amount of diacetate is at least 4 times that of the monoacetate, the concentration of the solutions being such that their content of combined acetic acid is equal to that of a diethylene glycol diacetate solution of concentration below 38%, removing the coagulating and regulating baths from their respective zones of operation, removing from said baths by distillation substantially all of the acetone derived from the spinning solution, adding to the baths sufficient diethylene glycol diacetate to restore their initial concentration of combined acetic acid while maintaining the amount of diethylene glycol diacetate in each bath at at least 88% of the total weight of diethylene glycol diacetate and monoacetate in the bath, and returning the baths to their respective zones of operation.

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 an acetone solution of the cellulose ester through a multi-hole spinning jet into a coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone and while allowing them to shrink freely, to the action of a regulating bath, the coagulating and regulating baths both being aqueous solutions selected from the group which consists of aqueous solutions of diethylene glycol diacetate and aqueous solutions of mixtures of diethylene glycol diacetate and monoacetate in which the amount of diacetate by weight is at least 4 times that of monoacetate, the concentration of the solutions being such that their content of combined acetic acid is equal to that of a diethylene glycol diacetate solution of concentration 30–35%, at least one of the baths containing both the diacetate and the monoacetate, removing the coagulating and regulating baths from their respective zones of operation, removing from said baths by distillation substantially all of the acetone derived from the spinning solution, adding to the baths sufficient diethylene glycol diacetate to restore their initial concentration of combined acetic acid while maintaining the amount of diethylene glycol diacetate in each bath at at least 88% of the total weight of diethylene glycol diacetate and monoacetate in the bath, and returning the baths to their respective zones of operation.

4. 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 an acetone solution of the cellulose acetate through a multi-hole spinning jet into a coagulating bath, stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone and while allowing them to shrink freely, to the action of a regulating bath, the regulating and coagulating baths both being aqueous solutions of a mixture of diethylene glycol diacetate and monoacetate in which the amount of diacetate is at least 4 times that of the monoacetate, the concentration of the solutions being such that their content of combined acetic acid is equal to that of a diethylene glycol diacetate solution of concentration 30–35%, removing the coagulating and regulating baths from their respective zones of operation, removing from said baths by distillation substantially all of the acetone derived from the spinning solution, adding to the baths sufficient diethylene glycol diacetate to restore their initial concentration of combined acetic acid while maintaining the amount of diethylene glycol diacetate in each bath at at least 88% of the total weight

of diethylene glycol diacetate and monoacetate in the bath, and returning the baths to their respective zones of operation.

5. 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 an acetone solution of the cellulose acetate through a multi-hole spinning jet into a coagulating bath at a temperature of 30–35° C., stretching the filaments during their passage through the coagulating bath, and then subjecting the filaments, until they are substantially free from acetone and while allowing them to shrink freely, to the action of a regulating bath at a temperature 8–15° C. below that of the coagulating bath, the coagulating and regulating baths both being aqueous solutions of mixtures of diethylene glycol diacetate and monoacetate in which the amount of diacetate is at least 88% of the total weight of diacetate and monoacetate and the concentration of the baths being such that their contents of combined acetic acid are substantially the same and are equal to that of a solution of diethylene glycol diacetate of concentration 30–35%, removing the coagulating and regulating baths from their respective zones of operation, removing from said baths by distillation substantially all of the acetone derived from the spinning solution, adding to the baths sufficient diethylene glycol diacetate to restore their initial concentration of combined acetic acid while maintaining the amount of diethylene glycol diacetate in each bath at at least 88% of the total weight of diethylene glycol diacetate and monoacetate in the bath, and returning the baths to their respective zones of operation.

6. Process according to claim 4, wherein the filaments are collected into an untwisted yarn and are stretched during their passage from the coagulating bath by means of tension applied to the yarn.

7. Process according to claim 4, wherein the number and dimensions of the holes in the spinning jet are so chosen that the yarn after it has shrunk has a denier above 500 while the individual filaments have a denier below 3.

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 an acetone solution of the cellulose acetate into a coagulating bath at a temperature of 30°–35° C., collecting the filaments into an untwisted yarn and applying to the yarn a tension

whereby 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 and while allowing it to shrink freely, to the action of a regulating bath at a temperature 8–15° C. below that of the coagulating bath, the coagulating and regulating baths both being aqueous solutions of mixtures of diethylene glycol diacetate and monoacetate in which the amount of diacetate is at least 88% of the total weight of the diacetate and monoacetate and the concentration of the baths being such that their contents of combined acetic acid are substantially the same and are equal to that of a solution of diethylene glycol diacetate of concentration 30–35%, and 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, removing the coagulating and regulating baths from their respective zones of operation, removing from said baths by distillation substantially all of the acetone derived from the spinning solution, adding to the baths sufficient diethylene glycol diacetate to restore their initial concentration of combined acetic acid while maintaining the amount of diethylene glycol diacetate in each bath at at least 88% of the total weight of diethylene glycol diacetate and monoacetate in the bath, and returning the baths to their respective zones of operation.

9. Process according to claim 3, wherein the filaments are subjected to the action of the regulating bath while on a thread-advancing thread-storage device.

10. Process according to claim 4, wherein the filaments are subjected to the action of the regulating bath while on a thread-advancing thread-storage device.

11. 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.

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