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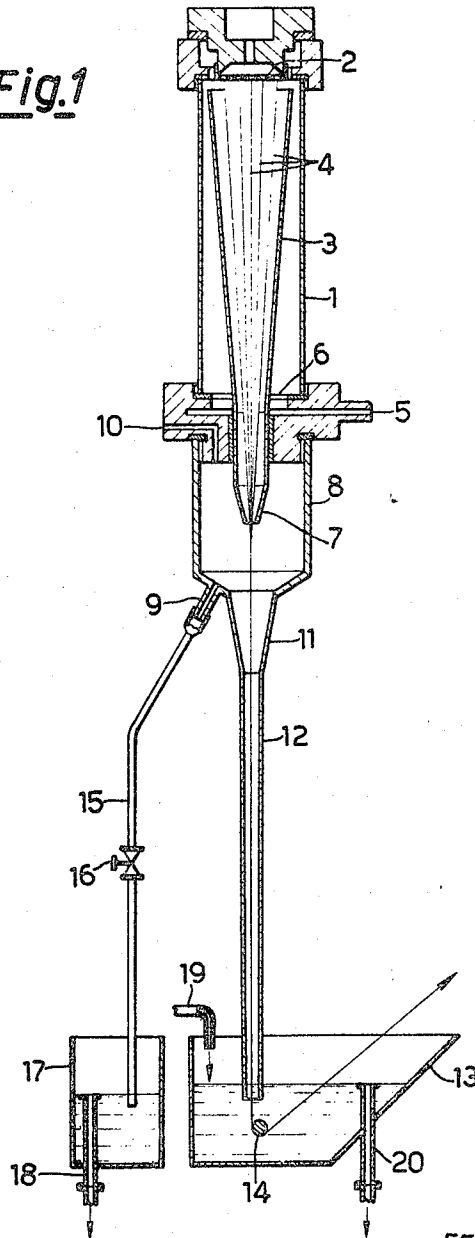
METHOD AND APPARATUS FOR THE PRODUCTION OF MANMADE FIBRES

AND MANMADE FIBRES OBTAINED THEREBY

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2 Sheets-Sheet 1

Fig. 1



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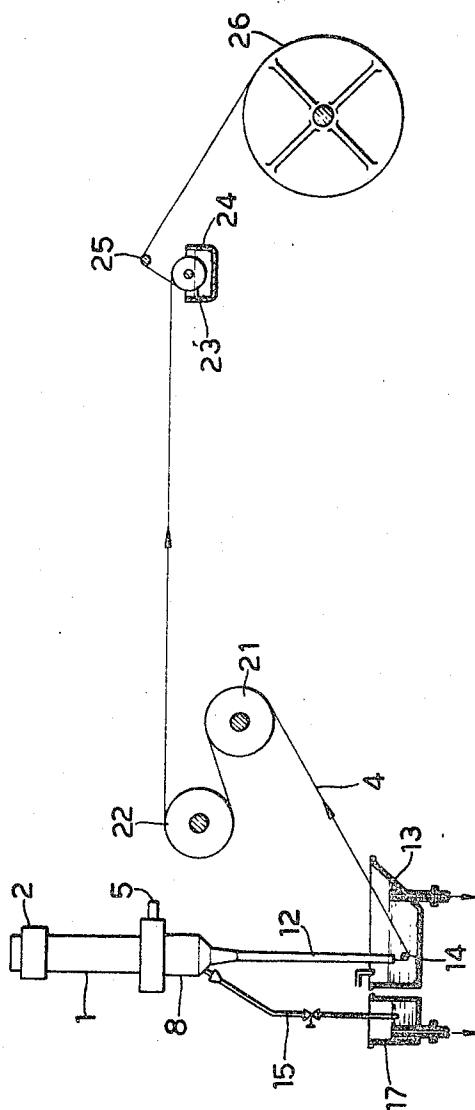
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2 Sheets-Sheet 2

Fig. 2



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1

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METHOD AND APPARATUS FOR THE PRODUCTION OF MANMADE FIBRES AND MANMADE FIBRES OBTAINED THEREBY

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12 Claims

ABSTRACT OF THE DISCLOSURE

A method and a device are disclosed for wet spinning cuprammonium regenerated cellulose wherein the extruded monofilaments are caused to contact the coagulation bath when they are still in a fluid, high-viscosity condition, the advantage being afforded that spinning rates much higher than those of the prior art for cuprammonium cellulosic yarns can be obtained, other advantages being those of readily obtaining low-denier filaments and almost completely removing the spinning water from the coagulation bath.

This invention relates to a method and an apparatus for wet spinning high-tensile filaments or staples based on cellulose which has been regenerated by a cuprammonium solution.

It is known that manmade fibres based on cellulose which has been regenerated from a cuprammonium solution, as produced with the conventional procedures, have a relatively poor wet tensile strength, in the order of magnitude of about 1.7 grms. per denier.

On the other hand, several methods have been suggested in order to improve this feature, which is of outstanding importance for the employment of the fibres in the textile art. Among these prior methods, the Italian patent specification No. 569,082 suggests the direct injection of the cuprammonium-treated cellulose solution, extruded in filamentary form through the holes of a spinneret, into a coagulating bath consisting of an aqueous solution of alkali metal salts (or alkaline earth metal salts), ammonia and ammonia-complexing substances, such as copper, zinc cadmium, cobalt and nickel salts. The thusly coagulated filaments are then strongly drawn and thoroughly regenerated in a mineral acid bath.

A shortcoming of the above method lies in that only limited spinning rates can be attained, e.g. in the order of 10–15 meters per minute, so as to prevent the filament from being damaged during progress of the subsequent mineral-acid regenerative step. This drawback apparently occurs in that the spinning solution, in filamentary form, contacts the coagulating bath immediately is extruded through the spinneret holes, that is to say, when it still retains a low-viscosity condition. An additional disadvantage of this method is that very low denier monofilaments, for example lower than 2 denier, cannot be obtained, whereas these could be extremely desirable in several textile applications.

2

According to another method, disclosed in the Italian patent specification No. 651,383, the filaments extruded through the spinneret are maintained within the spinning funnel in a fluid state and emerge therefrom with a viscosity which is considerably higher than that at the outlet of the spinneret and with an already partially ordained molecular configuration. Filaments in such a fluid state, or high-viscosity state, are treated with a protective solution of magnesium sulphate and concurrently subjected to the first draw. The optimum orientation thus achieved is then set with a quick-acting treatment with mineral acids.

The high-viscosity, or fluid, state of the filaments emerging from the spinning funnel is obtained by properly metering the rate of flow and the temperature of the spinning water. According to the Italian patent specification above mentioned, a fluid, or high-viscosity state should be intended as an incipient coagulation of the filament, unaccompanied, however, by any partial regeneration in proper sense, such as is experienced, conversely, already within the spinning funnel in the course of the conventional spinning process. The above mentioned Italian patent surmises that the high viscosity state in question may correspond to viscosities in the order of about 60,000 poises. Although reference will be made hereinafter to such a state, the present applicant intends it to be referred to viscosities of at least 15 times multiple of the original viscosity of the cuprammonium-cellulose solution.

It is thus possible, with the device disclosed in the Italian patent specification No. 651,383, to obtain higher spinning rates, for example in the order of magnitude of 30 to 40 meters a minute. The tensile strength ratings of the filament, however, were still unsatisfactory, mainly on account of their considerable degree of variability. In addition, the device in question does not allow an adequate separation of the spinning water from the protective solution.

Also the Swiss patent specification No. 312,219 discloses a method for producing high-tensile fibres based on cellulose which has been regenerated by a cuprammonium solution, wherein the filaments extruded through the spinneret emerge from the spinning funnel in a high-viscosity state, as high as 50,000–60,000 poises, which is defined as an optimum condition, to be then subsequently drawn. This last method, however, permits only limited spinning rates and, moreover, the tensile strength ratings of filaments produced in test runs have proven to be, in actual practice, only slightly over the conventional ones.

A method and an apparatus have now been found, which not only permit that very high wet tensile strength rating may be obtained for the cellulose filament, but, in addition, permit the attainment of hitherto unattained spinning rates, i.e. up to 110 meters a minute and over.

Another advantage afforded by the inventive device is due to the reduced bulk of the spinning assembly.

A further advantage of the inventive device and method lies in that they permit an almost complete separation of the spinning water from the coagulating bath.

A still further advantage of the inventive device and method lies in the fact that extremely low-denier monofilaments, under 2 denier, can be obtained.

The foregoing and other advantages will become more clearly apparent hereinafter.

The inventive device for wet spinning high-tensile filaments or staples based on cellulose which has been regenerated from a cuprammonium solution, is characterized by the following component parts, listed in downstream sequential order: a spinning chamber containing an extrusion head for extruding the solution through a spinneret and a conical spinning funnel located beneath said spinning head, a negative-pressure chamber integral with the spinning chamber and having a siphon discharge for the spinning water, the end portion of the spinning funnel opening into said negative-pressure chamber, a rectilinear tubing united to the negative-pressure chamber by a conical fitting whose length and inside diameter are in a ratio comprised between 60 to 1 and 20 to 1, but preferably between 40 to 1 and 30 to 1, a tub positioned below the rectilinear tubing so that the tub edge may lie at a level above that of the lower end of said rectilinear tubing.

The inventive method for wet spinning high-tensile filaments or staples based on cellulose which have been regenerated from a cuprammonium solution and in which the filaments are extruded from a spinneret, drawn down through a spinning funnel under the action of a water jet, emerge from the funnel in a still fluid, or high-viscosity, state, to be subsequently coagulated in such a condition in a bath of alkaline earth metal salts, is characterized in that the method is carried out by the device described above, so that the filaments extruded through the spinneret come down along the spinning funnel under the drawing action determined by a downwardly directed water flow, are then passed into a negative-pressure chamber from which the spinning water is siphoned out, and in which the filaments contact, in a still fluid, high-viscosity condition, a coagulation bath consisting of an aqueous solution of an alkaline earth metal salt, copper ions and ammonia, go through the coagulation bath along the rectilinear tubing in a comparatively relaxed condition, meeting a progressively increasing concentration gradient, referred to the alkaline earth metal salt so that, as the filaments contact said coagulation bath in the negative-pressure chamber, the concentration of the alkaline earth metal salt lies between 0.05 molar and 0.3 molar, whereas at the discharge from said rectilinear tubing, whose lower end is immersed in the coagulating bath contained in the tub, said concentration is more than 1-molar and preferably is 2-molar.

The filament is then withdrawn from the coagulation bath by one or more drawing rollers. The tractive force of said rollers must be such, also relatively to the spinning rates, that the tension of the filament as it emerges from the bath is preferably between 5 and 15 g. Thus, a basic feature of the inventive method provides that along the spinning funnel the filaments undergo a draw which is essentially determined by the entraining force of the downwardly directed spinning water flow, whereas at the discharge from the spinning funnel the filaments are to pass to a condition of comparative relaxation and, in this latter condition, are passed along the first portion of the coagulating bath along the upper portion of the rectilinear tube where they meet a progressively increasing gradient of concentration of the coagulating bath.

Said condition of comparative relaxation, which encourages the progressive action of the coagulating bath on the filaments, is obtained inasmuch as the tractive force of the downwardly directed spinning water flow is lacking, said water being then discharged almost completely from the negative-pressure chamber through the siphoning system, whereas the coagulating bath is nearly at standstill along the rectilinear tubing.

In the second portion of the rectilinear tubing, the tractive force of the drawing rollers, placed downward from the coagulating bath, progressively unfolds its action, by tensioning the filaments, so that, at the outcome from the bath, they have a tension of 5-15 g. It is obvious that

in the second portion of the rectilinear tubing the progressive tension state of the filaments is obtained at the expense of a slight draw of the filaments, the latter having been plasticized by the coagulation bath. The magnitude of said preliminary draw should not exceed 5%, however, whereas the final draw of the filament, which is generally of 100-150% is made past the stretching rollers by means of one or more drawing rollers which are rotated at a speed which is higher than that of the stretching rollers. Upon completion of drawing, the filaments are thoroughly regenerated in a mineral acid bath, for example by causing them to pass over an idle roller dipping in the bath. Hydrochloric acid is generally the preferred mineral acid.

On completion of the acidic regeneration, the thread can be wound on a reel or any other proper winding member, or converted into staples. It should be observed, however, that the final draw of the thread is not essential to the ends of the present invention. A process without final draw could also be performed. If so, the tensile strength rating of the resultant thread would be at least 15% less than that obtainable by making the final draw, but still sufficiently high. On the other hand, an elongation at break of more than 10% could be obtained, which might be desirable for certain uses of the thread.

Another characteristic feature of the inventive method is that the filaments enter contact with the coagulation bath, not only in a state of comparative relaxation, but also in a high-viscosity condition, without experiencing, however, any partial regeneration of the filaments. The high-viscosity state is obtained by properly adjusting the rate of flow of the spinning water stream and controlling the temperature thereof at not over 45° C. and preferably close to the ambient air temperature.

The coagulation bath comprises an aqueous solution which contains, dissolved therein, an alkaline earth metal salt, copper ions and ammonia. To the ends of the present invention, calcium chloride is the preferred alkaline earth metal salt.

The calcium chloride concentration in the bath held by the tub is higher than 1-molar and, preferably, is 2-molar. The bath is also supplemented by Cu^{++} ions in the preferred concentration, which is from 0.2 to 2 grms. per liter and by ammonia, the latter being, with respect to the Cu^{++} ions, in a weight ratio between 1/2 and 1/8.

Along the rectilinear tubing a concentration gradient is established, which has been indicated in the present disclosure as being referred to calcium chloride, but which should also be intended, in the correct proportions, as applicable to the cupric ions and ammonia.

The concentration gradient in the coagulating bath along the rectilinear tubing is rapidly set up at the beginning of the process run and is originated by the whirling mixing motion of the spinning water stream coming down from the spinning funnel with the coagulating bath stream which is ascending along the rectilinear tubing.

Once the desired equilibrium conditions have been set up, the spinning water is discharged, as a major fraction, through the siphon of the negative-pressure chamber, along with a minor fraction of the coagulation bath constituents. It is, however, practicable, if so desired, to achieve a complete separation of the spinning water from the coagulating bath by appropriately proportioning the length of the rectilinear tubing and the rate of flow through the siphon. It has been seen, however, that a small fraction of the coagulation bath constituents is dumped with the spinning water by the siphoning system.

The temperature of the coagulating bath contained in the tub is preferably comprised between 15° C. and 30° C.

The invention will now be illustrated in more detail with reference to the accompanying drawings which, however, should not be construed as limitations.

In the drawings:

FIGURE 1 is a front elevational view, partly in section, of the spinning device, and

FIGURE 2 is an overall view of the spinning device and the thread-collecting assembly.

Having reference to FIG. 1, the numeral 1 indicates the spinning chamber in whose interior a spinning funnel 3 is positioned. Filaments, such as 4, are extruded from the spinning head 2, through the holes of a spinneret. The spinning water completely fills the inside of the spinning chamber, being fed through the inlet duct 5 and coming up, through an annular port 6, alongside the outer walls of the spinning funnel, to straddle then into the interior of said funnel, wherefrom it goes down and reaches the negative-pressure chamber 8 through the end portion 7 of the spinning funnel.

The negative-pressure chamber 8 is integral with the spinning chamber 1. The negative-pressure chamber 8 has a frustoconical bottom portion which, through the conical fitting 11, communicates with the rectilinear tubing 12. The bottom portion of the rectilinear tubing dips in the bath contained in the tub 13. A major fraction of the spinning water is discharged through the discharge duct 9 along the tube 15, the latter fitted with an adjustable cock 16. The lower end of the tube 15 dips in a container 17 from which the spinning water is discharged through the overflow tube 18. The level of the liquid contained in the container 17 can be adjusted by adjusting the level of the overflow tube 18. The latter is adjusted so that the level of the spinning water collected in the container 17 is below the level of the coagulation bath contained in the tub 18. The level of the coagulation bath in the tub 13 is adjusted, in turn, by the overflow tube 20. The coagulation bath, discharged through the overflow tube 20, is recycled upon filtration and restoration to its initial concentration and fed into the tub 13 again, through the duct 19.

By virtue of the two different levels of the liquids contained in the container 17 and in the tub 13, a siphon is obtained, whose operation is determined by the negative-pressure chamber 8.

Due to the siphon effect, the coagulation bath ascends along the rectilinear tubing 12, to be gradually admixed with the spinning water coming down from the spinning funnel.

The following procedure is adopted to initiate the siphon when starting the spinning operations.

The spinning head 2 is unscrewed, the cock 16 stopped and the lower end of the rectilinear tubing 12 plugged. The flow of spinning water is then set up by a cock (not shown in the drawings) so that the water completely fills the rectilinear tubing, the negative-pressure chamber and the spinning chamber. During this operation, the vent 10 is kept open until the ascending water has filled the negative-pressure chamber completely, whereupon the vent 10 is closed and the topping up of the spinning chamber is completed. As the water level reaches the level of the spinning head, the flow is discontinued and the spinning head is screwed in its position. On completion of this step, the spinning of the cuprammonium cellulose solution is begun through the spinning head while simultaneously restarting the spinning water flow. The lower end of the rectilinear tubing 12 is reopened, the same being true of the vent 10, so that water begins to flow out of the negative-pressure chamber.

As the liquid level in the negative-pressure chamber has fallen to about one half of the height thereof, the vent 10 is closed again. Meanwhile, the filaments extruded through the spinning head have already come down along the rectilinear tubing. Due to the vent 10 being closed, the liquid column along the rectilinear tubing 12, the conical fitting 11 and the negative-pressure chamber 8, up to about one half of the height thereof, is interrupted. To cause the coagulation bath to ascend along the rectilinear tubing 12, it will now be sufficient to open the cock 16

and slightly suck through the lower end of the tubing 15 and thus the siphon created by the level differential of the liquids contained in the tub 13 and the container 17, respectively, will become operative, and an appropriate initial adjustment of the cock 16 will permit a quick ascending movement of the coagulation bath along the rectilinear tubing 12 so as to drive off the water contained therein. Subsequently, the cock 16 will be suitably adjusted so that the major fraction of the spinning water may be dumped through the duct 9 along with small fractions of the coagulation bath.

Referring to FIG. 2, the thread enters the coagulation bath held in the tub 13, is then withdrawn by the stretching rollers 21 and 22, with the thread being deflected by passing over the idler roller 14. The thread is then collected by the roller 23 whose surface speed is greater than that of the rollers 21 and 22, the desired draw of the thread being thus accomplished. The lower portion of the roller 23 dips in the bath contained in the tub 24, which contains a diluted mineral acid. On completion of a whole revolution about the roller 23, the thread is passed over the rod 25 and wound on the reel 26.

EXAMPLE 1

A cuprammonium solution containing 9.3% cellulose, 4.1% copper and 7% ammonia, fed with a gear pump to the spinning head, was extruded through the holes of a spinneret having 45 holes, each of a diameter of 0.8 mm. The spinning funnel was 450 mm. long whereas the rectilinear tubing had a length of 520 mms., with a diameter of 10 mms. The winding speed of the thread on the winding member (spinning rate) was 80 meters a minute. The final count of the yarn was 60 deniers.

The spinning water rate of flow was 150 cu. cms. per min., the water temperature being 19° C.

The coagulation bath contained 270 grms. per liter of bihydrated calcium chloride, 1 gm. per liter of copper ions, and 3.5 grms. per liter of ammonia. The sp. gr. of the liquor was then 1.160 at 20° C. and its temperature was kept at 21° C. The spinning water was discharged from the negative-pressure chamber at a rate of flow of 160 cu. cms. per min. and its sp. gr. was 1.020 at 20° C. The calcium chloride concentration in the discharged water was 24 grms. per liter. The final draw of the thread between the stretching rollers and the drawing roller took place with a drawing ratio of 2.05.

The textile specifications of the thread, as measured with an Istron tester according to the BISFA standards were as follows:

Conditioned tensile strength	-----grs./den..	4.3
Wet tensile strength	-----grs./den..	3.4
Conditioned elongation	-----percent..	8
Wet elongation	-----do.....	8.5
Wet modulus of elasticity	-----grs./den..	32

The same textile specifications, as determined on individual component monofilaments of the thread gave, conversely, the following values:

Conditioned tensile strength	-----grs./den..	4.5 to 5
Conditioned elongation	-----percent..	8.5

EXAMPLE 2

A spinning operation was repeated under the same conditions as in Example 1, the only difference being that the discharge of the spinning water was made at a rate of 150 cu. cms. per minute. In this case, the sp. gr. of the discharged spinning water was 1.010 at 20° C., whereas the calcium chloride concentration in the same water was 10 grs. per liter. The textile specifications were as follows:

Conditioned tensile strength	-----grs./den..	4.1
Wet tensile strength	-----grs./den..	3.1
Conditioned elongation	-----percent..	8
Wet elongation	-----do.....	8.5
Wet modulus of elasticity	-----grs./den..	28

EXAMPLE 3

A spinning operation was repeated with very much the same conditions as in Example 1, the only difference being that the discharge of the spinning water was made at at rate of 140 cu. cms. per min., so that the sp. gr. of the discharged water became 1.006 at 20° C. The concentration of calcium chloride was reduced to 3 grs. per liter. The textile specifications of the thread were as follows:

Conditioned tensile strength	-----grs./den--	3.9
Wet tensile strength	-----grs./den--	3.0
Conditioned elongation	-----percent--	8.3
Wet elongation	-----do-----	8.8
Wet modulus of elasticity	-----grs./den--	24

It is apparent, on the basis of the examples given, that it is possible to achieve a complete separation, from the coagulation bath, of the spinning water discharged from the negative-pressure chamber, by merely adjusting said discharge.

It is apparent, however, that a really complete separation is not advisable, in that the optimum values of the textile specification, especially the tensile strength, are obtained when a slight amount of calcium chloride is discharged with the spinning water.

EXAMPLE 4

A cuprammonium solution, containing 9.2% cellulose, 4.0% copper and 7.1% ammonia, is extruded through the holes of a spinneret having 45 holes, of a diameter of 0.8 mms. each.

The spinning funnel had a length of 550 mms. whereas the rectilinear tubing was 520 mms. long and had a diameter of 100 mms. The speed at which the thread was collected on the winding member was 110 meters per minute and the final count of the yarn was 60 deniers once again. The rate of flow of the spinning water was 150 cu. cms. per minute, the water temperature being 23° C. The coagulation bath contained 270 grs./liter of calcium chloride (bihydrate), 1 gr./liter of copper ions and 3.5 grs./liter of ammonia.

The sp. gr. of the liquor was 1.160 at 20° C., its temperature being maintained at 21° C.

The spinning water discharged from the negative-pressure chamber flowed at a rate of 170 cu. cms. per minute and its sp. gr. was 1.026 at 20° C. The concentration of calcium chloride in the discharged water was 36 grs./liter.

The final draw between the stretching rollers and the drawing roller took place according to a draw ratio of 2.14.

The textile specifications of the yarn were as follows:

Conditioned tensile	-----grs./den--	4.1
Wet tensile strength	-----grs./den--	3.2
Conditioned elongation	-----percent--	8.1
Wet elongation	-----do-----	8.6
Wet modulus of elasticity	-----grs./den--	30

While the inventive device and method have been described herein in connection with spinning of a high-tensile thread of cuprammonium regenerated cellulose, the device can be applied also to the production of a thread of cuprammonium regenerated cellulose having the usual tensile values whenever a mineral acid bath is substituted for the coagulation bath. If so, a considerable simplification of the conventional process would be the result, along with advantageous characteristics for the produced thread, which would possess an improved regularity.

Generally speaking, the inventive device can be used whenever it is desired to achieve a separation of the spinning liquid and a bath which contains products having a specific action upon the thread.

In addition, the inventive device can be applied to the production of high-tensile cellulose staples, made of cuprammonium regenerated cellulose, with the concurrent

use of conventional technical means which are readily available to those skilled in the art and thus are not described in detail herein.

What is claimed is:

1. A method for wet spinning high-tensile filaments or staples based on cellulose which has been regenerated from a cuprammonium solution in which filaments or staples extruded from a spinneret come down through a spinning funnel under the cation of a water jet, emerge from the funnel in a fluid, or high-viscosity state and subsequently coagulated in said condition in a bath of alkaline earth metal salts, characterized in that the filaments or staples extruded through the spinneret pass through the spinning funnel under the drawing action determined by a downwardly directed water flow, pass into a negative-pressure chamber from which the spinning fluid is siphoned off, and in which the filaments or staples contact in their still fluid, high-viscosity condition, a coagulation bath consisting of an aqueous solution of an alkaline earth metal salt, copper ions and ammonia of relatively low concentration, and then while in a comparatively relaxed condition contact said coagulation bath having a progressively increasing concentration gradient, the concentration of said bath, based on the alkaline earth metal salt, ranging from between 0.05 molar and 0.3 molar at initial contact to a concentration of more than one molar at final contact.

2. A method according to claim 1, characterized in that calcium chloride is preferably used as the alkaline earth metal salt.

3. A method according to claim 1 characterized in that the concentration of the copper ions is in the coagulation bath at final contact is from 0.2 grs./liter and 2 grs./liter.

4. A method according to claim 1 characterized in that the copper ions and the ammonia in the coagulation bath on a weight ratio at final contact is from 1:2 and 1:8.

5. A method according to claim 1 characterized in that the filaments are subjected to a partial draw in the coagulation bath.

6. A method according to claim 1 characterized in that the final draw of the filaments is carried out past the coagulation bath.

7. A method according to claim 1 characterized in that the final draw of the thread is from 1 to 3 times the length of the thread.

8. A method according to claim 1 characterized in that the complete regeneration of the filaments by a mineral acid bath is made on a drawn thread.

9. A method according to claim 1 characterized in that the mineral acid for the complete regeneration of the filaments is hydrochloric acid.

10. A yarn based on cellulose which has been regenerated from a cuprammonium solution, as produced according to the method of claim 1.

11. A staple based on cellulose which has been regenerated from a cuprammonium solution, as produced according to the method of claim 1.

12. A device for wet spinning high-tensile filaments or staples based on cellulose which has been regenerated from a cuprammonium solution comprising a spinning chamber containing an extrusion head for extruding the solution through a spinneret and a conical spinning funnel located beneath said spinning head; a negative-pressure chamber integral with the spinning chamber and into which the end portion of the spinning funnel opens; a first rectilinear tubing united to the negative-pressure chamber by a conical fitting whose length and inside diameter are in a ratio between 60:1 and 20:1 and preferably being between 40:1 and 30:1, a tub placed beneath said first rectilinear tubing; a second rectilinear tubing extending from said negative-pressure chamber and axially with said spinning chamber, and a second tub placed beneath said second rectilinear tubing so that the edge of

the tub is at a level higher than that of the lower end of said second rectilinear tubing, said elements being constructed and arranged in order that the major amount of spinning fluid from said negative-pressure chamber is drawn off through said first rectilinear tubing, that fluid from said second tub passes up said second rectilinear tubing to said negative-pressure chamber, whereby filaments or staples being drawn off from said negative-pressure chamber pass into said second rectilinear tubing along with only minor amounts of spinning fluid and contact fluid from said second tub before being exhausted into said second tub at a level below the fluid level of said tub.

5

10

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