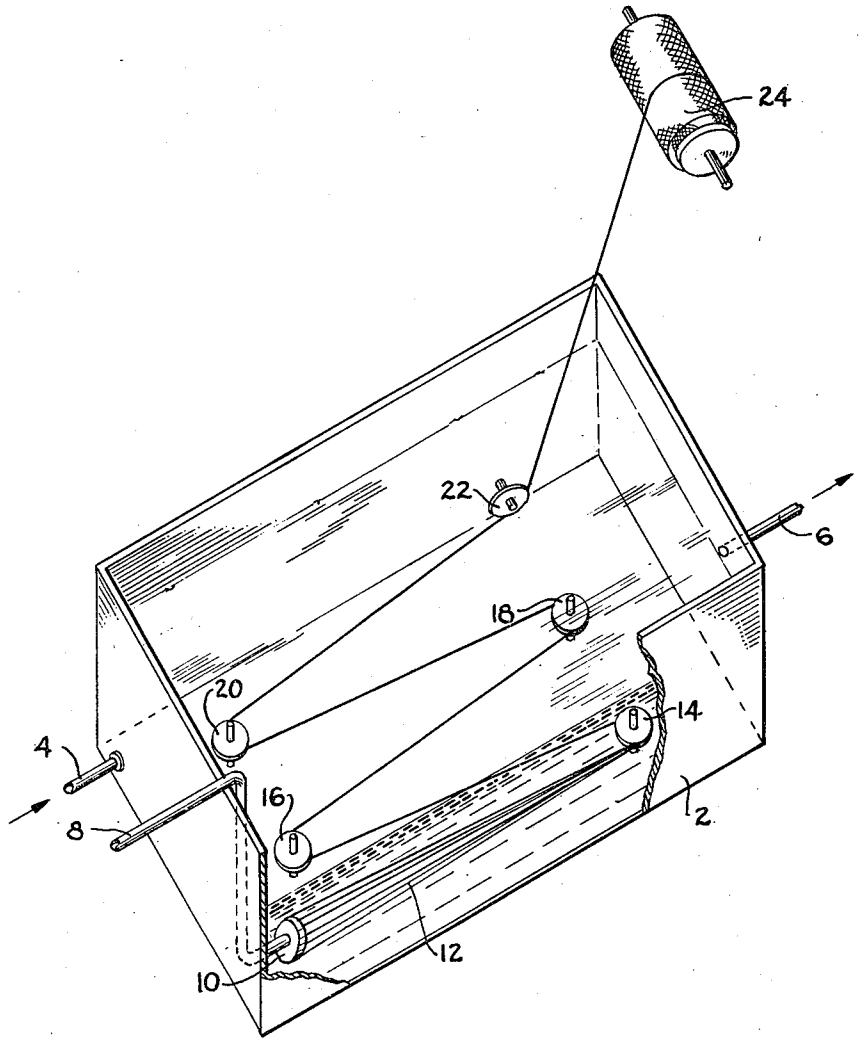
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METHOD OF SPINNING ARTIFICIAL FILAMENTS

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METHOD OF SPINNING ARTIFICIAL FILAMENTS

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This invention relates to an improved method for the spinning of viscose rayon yarn, i. e., regenerated cellulose yarn produced by the viscose process. More particularly, it relates to a method whereby viscose rayon yarn possessing satisfactory physical properties may be produced at spinning speeds greatly in excess of those conventionally used in the arts.

A considerable portion of the cost of viscose rayon yarns is due to the large investment in the spinning machines and to the cost of labor involved in the operation of these machines. Any alteration in the process which permits increasing the spinning speed and consequently the production of a greater poundage of yarn from a given number of machines will result in a decreased cost of production. This fact has been well recognized in the art and a study of the literature shows many proposals to this end. As a result of improvements and refinements in the art the maximum permissible spinning speed has been gradually increased from less than 1000 to something over 3000 inches of yarn per minute.

Such increase in spinning speed has been made possible by the development of improved equipment including spinning machines and bath guides, and by the development of improved materials such as wood pulps of improved characteristics. In the main, this increase has been achieved in a series of small steps involving the application of these improvements together with small changes in the various spinning factors such as viscose and bath composition, spinning tension, and length of travel in the coagulating bath.

Such changes have not only made possible an increase in the speed of spinning but they have also operated to yield a yarn of greatly improved physical properties. Both factors have contributed greatly to the present increased use of rayon yarn.

However, despite these improvements and changes, recent attempts to further increase the speed of spinning have all had an adverse effect on the physical properties of the yarn. Increasing the spinning speed of a yarn, other conditions remaining the same, results in a decrease in the tenacity of the yarn in the dry state (dry tenacity) as well as a decrease in the elongation of the yarn in the dry state (dry elongation). The "quality factor" (dry tenacity × dry elongation) of the yarn consequently decreases very markedly. The extent of this adverse effect becomes larger as the absolute speed of spinning is increased so that, in the light of present day knowledge, spinning speeds much above 3500 inches per minute are impractical.

It is an object of this invention to provide an improved method for the spinning of viscose rayon yarns.

It is a more specific object of this invention to provide an economical means of spinning a viscose rayon yarn of good and satisfactory quality at spinning speeds greatly in excess of those conventionally used in the commercial processes.

Other objects of the invention will appear hereinafter.

The invention is based on the discovery that a viscose rayon yarn possessing satisfactory physical properties may be spun at a greatly increased thread speed by maintaining the coagulating bath at an elevated temperature.

The present invention relates to the spinning of yarn at high linear speeds; i. e., speeds of 5000 inches per minute and more as, for instance, 7000 inches per minute, and is meant to include speeds of 10,000 inches per minute and even higher. The term "elevated temperature" relates to temperatures of 60° C. and higher as, for instance, 85° C., and includes temperatures up to 100° C. and even higher.

The present invention is illustrated in the accompanying drawing in which Figure 1 is a perspective view showing an apparatus suitable for use in carrying out the invention.

Referring to the drawing, reference numeral 2 designates a container for a coagulating, regenerating bath containing inlet conduit 4 and outlet conduit 6 whereby the temperature of a liquid contained in the bath may be maintained constant. A viscose solution is passed into the bath by means of conduit 8 and extruded within the bath through spinneret 10. The filaments 12 extruded from the spinneret are passed through guides 14, 16, 18, 20 and 22. From the guide 22, the yarn is passed from the bath and wound on to a package 24, such as a bobbin package.

As mentioned above, it has long been desired to spin viscose rayon yarn at the greatly increased thread speed with which this invention is concerned. However, all attempts to spin yarn at these speeds have been in baths of normal temperature and have yielded yarn of poor physical properties. In accordance with the principles of this invention, yarns of good physical properties are produced at these increased thread speeds by the use of a coagulating bath heated to a temperature of 60° C. and higher.

As above stated the quality factor of a yarn is the product of the dry tenacity and dry elonga-

tion of the particular yarn under consideration and constitutes a convenient measure of the physical properties of the yarn. A high quality factor indicates good physical properties. It is known that the tenacity and elongation of a yarn can be changed in opposite directions by changing the tension at which the yarn is spun and, if this change is not too great, the product of the two properties will remain approximately constant. For the data presented herein those yarns possessing the same quality factor could have been prepared with substantially identical properties by merely effecting a slight change in the tension employed during the spinning of these yarns; hence, the product indicated by the term "quality factor" provides a simplified means of expressing the physical properties of a yarn.

The reasons underlying the decrease in the physical properties of yarn spun in normal temperature baths as the spinning speed is increased are not entirely known nor understood. It was thought at one time that the yarn spun at these increased speeds did not have sufficient time to become completely regenerated. However, the decrease in the desired yarn properties was still obtained even when the bath travel was increased to a point where the yarn was permitted to spend as much time in the bath at high speed as at low speed. It is possible that the decrease in yarn properties can be overcome by a suitably different distribution of stretch in the yarn spun at high speed either with or without the use of longer bath travel. However, special friction guides, designed to equalize the stretching effect of the tension imposed on the yarn spun at high speed in the baths of various lengths, did not overcome this decrease in the desired physical properties. A third hypothesis was based on the theory that the rates of diffusion, coagulation and regeneration suitable for low spinning speeds were not suitable for use with high spinning speeds. These rates have been altered by increasing the acidity of the bath, by reducing the sodium sulfate content of the bath and by reducing the alkalinity of the viscose solution. However, in no case could yarns, having satisfactory physical characteristics, be obtained at high spinning speed; hence it would appear that the rates of diffusion, coagulation or regeneration as affected by the aforementioned factors of bath acidity, sodium sulfate content of the bath, and alkalinity of the viscose solution are not at fault. Neither does increasing the bath travel to increase the time for regeneration nor using special friction guides to change the distribution of stretch during the spinning operation affect this adverse result which is obtained as the spinning speed is increased.

It has now been found possible to spin a yarn having satisfactory physical characteristics at high speed when the coagulating bath is heated to a temperature of over 60° C. The basic reason underlying this discovery is not entirely known nor understood; however, it is apparently related, at least in part, to a particularly favorable condition of coagulation resulting from the high temperature per se and is not due merely to an increased rate of diffusion and reaction.

Such a conclusion is substantiated by the following experimental evidence. When yarn is spun at high speed into a bath maintained at 45° C. and is then immediately passed into a bath heated to 85° C., the improvement in yarn properties noted in the practice of the present invention is not obtained. Nor is it obtained even when the yarn travel in the initial 45° C. bath is reduced to less than one-half inch. This is in spite of the fact that the diffusion of the bath within the yarn filaments has just begun during this short travel through the cool bath and, save for the very outside of the individual filaments, no regeneration has taken place. In fact, experiments show that less than 2% of the cellulose xanthate can be regenerated in this time. Therefore, substantially all of the diffusion of the bath and regeneration of the yarn must take place during the travel of the yarn through the heated portion of the bath and the rates of these actions must be those characteristic of the high temperature. Nevertheless, the yarn is practically the same as if it had been spun entirely in a bath of normal temperature.

On the other hand, when a yarn is spun into a bath maintained at 85° C., and then, after less than one inch of travel in this bath is immediately passed into a bath of normal (45° C.) temperature, that yarn has substantially the improved properties obtained when the entire bath is heated to 85° C. In this case, the diffusion and regeneration take place largely at normal temperatures and the rates are those characteristic of the low temperature baths; yet the yarn resembles most closely those yarns that are spun throughout in a high temperature bath.

These experiments, together with the fact that merely increasing the rate of diffusion and regeneration does not give the desired result, point to the fact that the improved results obtained by the practice of this invention are due to some phenomenon that is not predictable from theoretical consideration and is further probably specific for viscose coagulation.

Still further evidence pointing to the existence of some such peculiar type of coagulation lies in the fact that cuprammonium viscosity tests show that yarns spun in accordance with this invention are less degraded than are yarns spun according to conventional commercial practice; i. e., at conventional speeds and temperatures. This fact is quite surprising and further suggests a peculiar type of coagulation in the case of yarns spun at high speed in a high temperature bath.

Further evidence pointing to the existence of a cooperative relationship between the temperature of the spinning bath and the speed of spinning is found in experiments conducted to determine the ability to spin yarn at various speeds and under a variety of conditions. This evidence relates to a factor that may be termed "spinning quality," and is closely related to such features of the spinning operation as the occurrence of broken filaments within the yarn, the frequency with which these broken filaments form wraps about the yarn guides and the rapidity with which the holes of the spinneret become plugged. Thus, this factor of "spinning quality" differs from the previously mentioned "quality factor" which is concerned only with the physical properties of the finished yarn.

This evidence indicates that, other conditions being the same, there exists for each spinning speed a minimum bath temperature below which it is impossible to spin yarn successfully, and the evidence further indicates that this minimum temperature increases as the speed of spinning is increased. For example, the following evidence is obtained in the spinning of yarn of 150-denier, 40-filament count, by the extrusion of a solution of viscose prepared from cotton linters and containing 7% cellulose and 6% sodium hydroxide

into a spinning bath containing 11% sulfuric acid, 19% sodium sulfate, 4% glucose and 0.7% zinc sulfate.

The spinning quality is excellent at a speed of 3300 inches per minute when the bath is heated to 35° C. or more.

The spinning quality is excellent at a speed of 4400 inches per minute when the bath is heated to a temperature of 45° C. or more.

It is impossible to spin yarn at a speed of 5400 inches per minute unless the bath is heated to a temperature of 55° C. The ease with which yarn can be spun at this speed is increased as the bath is heated to 65° C., but the spinning quality is not excellent until the bath temperature is raised to 75° C.–85° C.

It is impossible to spin yarn at a speed of 8000 inches per minute unless the bath is heated to 90° C. and here again the spinning quality is greatly improved by heating the bath to still higher temperature.

It is true that spinning quality and indeed the ability to spin yarn at all, is closely related to the operating conditions employed and changes in such factors as the composition of the viscose solution or coagulating bath, or the denier or filament count of the yarn being spun will affect the minimum bath temperature that can be used in the spinning of yarns at various speeds. Therefore, the exact values of the minimum and optimum temperatures given above apply only to the exact spinning conditions used in the experiments, as stated above. Nevertheless, it is generally true that for each spinning speed there is a minimum bath temperature below which it is impossible to spin yarn and it is further true that, other conditions being constant, this minimum temperature increases as the speed of spinning is increased. The existence of this relationship of spinning speed and bath temperature is quite surprising, and taken together with the relationship previously mentioned in connection with the quality factor of yarns spun at high speeds, points even more strongly to the theory that a peculiar and hitherto unnoticed type of viscose coagulation exists in the case of yarns spun at high speed in a bath heated to an elevated temperature.

It should also be noted in this connection that another important advantage closely associated with spinning quality and accruing from the practice of this invention is a substantial increase in the breaking tension of the yarns so spun. The breaking tension of a yarn is that tension at which a yarn will break during the spinning operation. For good spinning performance it is necessary that the breaking tension be somewhat greater than the spinning tension and it is desirable that a yarn possess as high a breaking tension as possible. Not only will an increased breaking tension permit a higher spinning tension to be employed so that yarns of higher tenacities can be spun, but if the same low spinning tension is used, it will tend to improve the spinning quality by virtue of the greater difference between spinning tension and breaking tension. Thus, there will tend to be fewer broken filaments in the yarn so produced.

The breaking tension of a 150-denier, 40-filament yarn spun form a solution of wood pulp viscose containing 7% cellulose and 6% sodium hydroxide and having a salt index of 4.0 at a speed of 3300 inches per minute, at 45° C., in a bath containing 11% sulfuric acid, 19% sodium sulfate, 4% glucose and 0.7% zinc sulfate, is about 40 to 45 grams. If the spinning speed is now increased to 5400 inches per minute, it is found that the breaking tension falls to a point below the spinning tension, so that it is impossible to spin yarn under these conditions. However, if, in accordance with the principles of this invention, the temperature of the spinning bath is increased as the spinning speed is increased, it is found that the breaking tension is substantially increased. For example, yarns spun at 5400 inches per minute in a bath heated to 78° C. possess a breaking tension of about 50 to 55 grams. Heating the bath to still higher temperatures gives even greater improvements in this factor. Naturally, the exact value of the breaking tension depends on the denier and filament count of the yarn and upon the conditions employed during the spinning operation.

The following examples will illustrate most clearly the improvements to be obtained by the practice of this invention, and will suggest a few of the preferred ranges of operation. These examples are not to be considered as limiting the scope of the invention.

Example I

Cotton linters viscose containing 7% cellulose and 6% sodium hydroxide and ripened to a salt index of 4.0 is spun into a bath containing 11% sulfuric acid, 21% sodium sulfate and 0.7% zinc sulfate, using a total bath travel of 25–30 inches and a spinning tension of 33, 43, and 47 grams, respectively, to give a yarn of 150 denier, 60 filaments.

Yarn	Spinning speed	Bath temperature	Tenacity (dry)	Elongation (dry)	Quality factor
	Inches/min.	*°C.*	*Grams/den.*	*Per cent*	
A	3400	45	1.85	20.8	38.4
B	5300	45	1.84	16.8	30.9
C	5300	85	1.98	18.3	36.2

Example II

Wood pulp viscose containing 7% cellulose and 6% sodium hydroxide and ripened to a salt index of 4.0 is spun into a bath containing 11% sulfuric acid, 19% sodium sulfate, 4% glucose and 0.7% zinc sulfate, using a total bath travel of 20 to 26 inches to give a yarn of 150 denier, 40 filaments.

Yarn	Spinning speed	Bath temperature	Spin tension	Tenacity (dry)	Elongation (dry)	Quality factor
	Inches/min.	*° C.*	*Grams*	*Grams/den.*	*Per cent*	
D	3,300	45	22.5	1.93	20.0	38.6
E	5,400	45	20–29	(1)	(1)	(1)
F	5,400	80	29	1.90	17.9	34.0

[1] It was impossible to spin yarn under these conditions.

This invention provides for the production, at greatly increased spinning speeds, of viscose rayon yarn of satisfactory properties by the use of a coagulating bath heated to an elevated temperature. The invention does not require the use of a special viscose solution nor does it require the use of coagulating baths of special composition, its advantages being realized by the use of the various conventionally used viscose solutions and coagulating baths known in the art. For example, viscose solutions containing from 6% to 8% cellulose, 4% to 8% alkali and substantially any degree of ripeness may be used. Similarly, coagulating baths containing the usual 7% to 15% sulfuric acid together with suitable

quantities of glucose, ammonium sulfate, sodium sulfate and, if desired, zinc, nickel or iron sulfate will be suitable. In the spinning of viscose rayon yarns according to the conventional bobbin and bucket processes, it is known that yarn may be spun with better results in baths of certain compositions than in baths of other compositions. It is also well known to vary the composition of the coagulating bath as the viscose solution is changed, either with respect to its concentration or composition. These well known expedients also hold in the spinning of acceptable yarn according to the present invention, and this invention contemplates the use of the most desirable bath composition for the spinning of yarn from any given viscose solution. Some of these desirable combinations are illustrated in the examples accompanying this specification.

In all cases, however, it is true that there exists an optimum temperature of the coagulating bath for each spinning speed employed. The exact value of this optimum temperature for any given spinning speed will vary slightly as the total denier of the yarn being spun or the composition of the viscose solution or coagulating bath is changed but, in accordance with the principles of this invention and as illustrated in the examples contained in this specification, the optimum temperature for yarns spun at a speed of 5000 or more inches per minute will be at least 60° C.

This invention is not limited to the spinning of viscose rayon yarns at low tension, such as tensions of 0.2 gram per denier. It may also be used in connection with much higher spinning tensions such as of the order of 0.7 gram per denier or even more. In this regard it might be well to note that this invention also possesses the important advantage of permitting a very accurate control of the spinning tension to be obtained. Tension is usually applied, during the spinning process, by causing the yarn to pass about one or more rotating guide rollers located within or without the bath. However, the liquid drag exerted on the yarn by the spinning bath also operates to develop tension and in the case of high speed spinning with which the invention is concerned, the effect of this liquid drag is so greatly increased that undesirably high spinning tensions are developed. It has now been found that when the bath is heated to an elevated temperature in accordance with this invention, the resulting lowered viscosity of the bath serves to reduce this liquid drag sufficiently to permit an accurate control of the tension applied to the yarn. This is a point of the utmost importance in the high speed spinning of a uniform, satisfactory yarn.

This invention has been discussed primarily in connection with the high speed spinning of yarns from viscose solutions which have been ripened to a normal degree and possess a common salt index of 4.0 or less. However, the invention is of equal if not greater importance in the high speed spinning of viscose rayon yarns from viscose solutions that have been ripened to a lesser extent and possess a common salt index of 5.0 or even higher as, for example, an index of 8.0. This is brought out most clearly by the following Example III which indicates the improvement in quality factor of yarns spun from viscose solution of high salt index and at high speed in accordance with the principles of this invention. The data contained therein illustrate most clearly the improvement in physical properties of these yarns as the temperature of the bath is raised above 60° C. In the past it has always been thought necessary to use a coagulating bath of special composition and maintained at a very low temperature when spinning yarn from viscose solutions of high index. Therefore, these results are of the utmost interest and importance.

Moreover, the improvements obtained by the practice of this invention are not limited to improvements in merely the quality factor of the yarns; the data of Example III show that this invention also provides a means of improving the softness of yarns spun from solutions of high index.

Example III

Wood pulp viscose containing 7% cellulose and 6% sodium hydroxide and ripened to the salt index indicated, is spun into a bath containing 11% sulfuric acid, 19% sodium sulfate, 4% glucose and 0.7% zinc sulfate using a bath travel of 20–25 inches and a spinning tension of 20–30 grams to give a yarn of 154 denier, 40 filaments.

Yarn	Salt index of viscose	Spinning speed	Bath temperature	Tenacity (dry)	Elongation (dry)	Quality factor	Softness
		Inches/min.	*°C.*	*Gms./den.*	*Per cent*		
G	4.0	3,300	45	1.93	20.0	38.6	96
H	4.0	5,400	45	(1)			
I	4.0	5,400	70	1.86	17.5	32.6	10
J	4.0	5,400	80	1.90	17.9	34.0	9
K	8.0	3,300	45	1.61	17.1	27.5	72
L	8.0	5,400	45	(1)			
M	8.0	5,400	70	1.98	18.9	37.4	105
N	8.0	5,400	85	2.03	20.9	42.5	99

[1] It was impossible to spin yarn under these conditions.

In accordance with the present invention, rayon yarn of acceptable physical properties can be spun at speeds of 5000 or more inches per minute.

The invention provides a method of spinning viscose rayon yarns which, according to cuprammonium viscosity tests, are less degraded than are viscose rayon yarns produced according to present known methods.

The invention provides a method whereby viscose rayon yarns of improved quality may be spun at speeds of 5000 inches per minute or more from viscose solutions that have been ripened to a very small extent and possess a comon salt index as high as 8.0. Furthermore, in accordance with the principles of this invention it is not necessary to use coagulating baths of special composition in the spinning of these yarns.

The invention provides a means whereby the spinning tension may be accurately controlled and undesirably large increases in spinning tension may be avoided as the speed of spinning is increased.

The invention further provides a means whereby the breaking tension of yarns may be substantially increased. This increase in breaking tension may be used either to permit the spinning of yarn of higher tenacity or to improve the spinning quality of the yarn by virtue of greater difference between spinning tension and breaking tension.

Although this discussion of the invention has been limited to the spinning of yarns from a viscose solution, it is obvious that the invention should not be so limited. Its principles can be used successfully in the manufacture of films, tubings, and other structures of regenerated cellulose from viscose solutions. Such articles, pre-

pared in accordance with the principles of this invention, will be found highly suitable for use in the art.

Since it is obvious that many changes and modifications can be made in the above-described details without departing from the nature and spirit of the invention, it is to be understood that the invention is not to be limited to these details except as set forth in the following claims.

I claim:

1. The process which comprises spinning a viscose solution at a speed of at least 5000 inches per minute into an acid coagulating bath containing from 7% to 15% sulfuric acid and having a temperature of at least 60° C.

2. The process which comprises spinning a viscose solution, containing from 6% to 8% cellulose and from 4% to 8% alkali, at a speed of at least 5000 inches per minute, into a coagulating bath containing from 7% to 15% sulfuric acid and having a temperature of at least 60° C.

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