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METHOD OF SPINNING LOW ELONGATION VISCOSE RAYON

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1 Claim. (Cl. 18—54)

The present invention relates to the manufacture of viscose rayon and more particularly to a process in which viscose is spun into an acid spinbath and subsequently highly stretched in a hot dilute acid bath to produce yarn of controlled low elongation.

It is, of course, well known that the strength of viscose yarn can be increased by stretching the same, preferably within a short time after it leaves the spanbath. And it is thought that the strength increases in direct proportion to the increase in stretch. The system generally used is known as the two-bath spinning process and includes a normal spinbath containing sulfuric acid and salts such as sodium sulfate and zinc sulfate, and a second weakly acidic bath which is maintained at a high temperature, for example, in the neighborhood of 90° C. and above.

In view of the fact that a very high strength is desirable in the production of some types of yarn, work has been done in view of finding ways of increasing the limit to which yarn may be stretched without filamentary breakage incurring. Along this line, Kayser in U.S. Patent No. 2,452,130, having common ownership with the present application, discovered that freshly spun viscose yarn may be given an unusually high stretch by incorporating formaldehyde in the spinning system. According to Kayser, the formaldehyde renders the yarn more stretchable and a yarn of high tensile strength may be produced.

However, for many end uses, it is preferred that the yarn have not only a high strength but also a low elongation. Although it is possible to reduce the elongation by increasing the stretch imparted to the yarn, it has been found that after a point the elongation may not be reduced by additional stretching. Where the elongation is undesirably high even by employing a high stretch, it has been the practice heretofore to give the yarn a final stretch in a wet condition. Thereafter, this yarn is dried under tension. However, upon being wet again, the yarn has a great tendency to shrink. This shrinkage is just as undesirable as too high an elongation in many industrial applications. Consequently, the need exists for a spinning process by which a high strength, low elongation yarn may be produced.

An object of the present invention is, therefore, to provide a process for increasing the stretchability and strength and for reducing the elongation of viscose yarns.

Another object of the present invention is to provide a new acid spinbath composition useful in producing viscose yarn having the aforesaid desirable properties.

These and other objects are accomplished in accordance with the present invention by spinning viscose into a low acid spinbath maintained at a low temperature and stretching the freshly formed yarn.

It is necessary according to the present invention to maintain the temperature of the spinbath within the range of 20° C. to 45° C. Above 45° C. the improvement of the present invention is not attained. Below 20° C. spinning difficulties arise. Furthermore, it is essential that the sulfuric acid content of the spinbath be adjusted to between 2 and 7%. Above 7% the increased stretchability and reduced elongation of the present invention are notably decreased. Below 2% acid many spinning

problems occur. However, for best results, it has been found that the preferred range of acid is between 4 and 6%.

It was very surprising that the advantages of the present invention could be obtained under the above mentioned spinning conditions, since normally it was thought that with a low acid spinbath a higher temperature should be used.

By employing a low temperature bath there are many attendant advantages. For example, less energy is consumed and the worker is exposed to less spinning bath vapors and does not have to work with hot acid.

However, the most important advantage resides in the fact that the threads produced in accordance with the invention have lower elongation and can withstand a higher spinning stretch than yarn obtained from normal spinning. Furthermore, this high stretch may be attained at a lower stretching tension. In fact, the stretchability of the freshly spun threads at the low spinbath temperature is so great, depending on the combination of viscose and spinbath variables, that it is possible to stretch the threads up to 100% and more in the one-bath process, which values were thought to be restricted to the two-bath process. Also, in the two-bath process the temperature of the second dilute bath may be kept lower than otherwise considered necessary, if the present invention is followed. For example, temperatures of 50° C. to 70° C. are sufficient in order to achieve the amount of stretch which formerly could be obtained at temperatures much higher than 70° C. in the second bath.

An additional important advantage of the process according to the present invention lies in the fact that it is possible to vary within limits the ratio between the breaking load and breaking elongation. It is known that with every spinning process the numerical value of this ratio can be somewhat modified by various changes in the spinning conditions. However, for given spinning conditions it is possible by the present invention to control the resulting elongation during the manufacturing of the articles to a greater extent than in normal spinning. Thus, there is more flexibility in adapting the yarn to meet the physical property requirements of the customer.

The present process has proved to be of particular value in those cases where yarns with different elongation values are to be spun from a common spinning bath installation to different spinning machines. By the present invention it is only necessary to adjust the bath temperature at the machines to different levels.

The present invention also contemplates spinning in the presence of an additive which modifies the regenerating-coagulating process of the viscose. These additives are referred to generally in the art as viscose modifiers. The yarn produced under the influence of a viscose modifier has many distinctive characteristics that distinguishes it from normally spun yarn. For example, cross sections of the filaments produced in a system employing a modifier when stained and viewed under a microscope in a well known manner reveal a skin-type of yarn having a periphery of notably reduced crenulations.

Many modifiers are known in the art today. Among these, the following specifically mentioned compounds have been found to be particularly satisfactory for use in the present invention: reaction products of polyalkoxy glycols with fatty amines, fatty alcohols, fatty acids, aromatic acids, alcohols or amines; aliphatic or aromatic monoamines, diamines or polyamines; quaternary ammonium bases or heterocyclic bases; monothiocarbamates or dithiocarbamates; and 2-mercaptobenzimidazole or mercaptothiazole.

The modifiers may be used in various amounts, depending upon the spinning conditions and the particular modifier employed. However, in general, the minimum

concentration for addition to the viscose is 0.02%. Preferably, the modifier is added in the amount of about 0.02 to 0.6%. When the additives are added to the spinbath, the preferred concentration is in the range of 0.1 to 5.0 g. per kg. of spinbath.

By using a modifier in the present invention, both the advantages of modified spinning and the advantages of using the low acid spinbath maintained at a low temperature may be obtained cumulatively. This is surprising in view of the fact that when certain modifiers are used threads of high elongation may result.

Yarns of extremely high strength can be produced by using viscose additives at spinbath temperatures lower than 50° C.; for example, yarns having strengths of 500 g./100 denier and higher (air-dry) may be obtained.

In copending application Serial No. 641,851 having common ownership herewith, there is disclosed a method of spinning viscose in the presence of a viscose modifier into a spinbath containing a small amount of formaldehyde. It has been found that the low acid and low temperature spinbath of the present invention further improves the yarn produced in accordance with the teaching of the application.

Besides the critical acid concentration of the spinbath, other components of the spinbath are important to the present invention. Ordinarily, it is required that the bath contain a zinc salt, preferably zinc sulfate in the amount of at least 2% by weight. Amounts as high as 15% zinc sulfate are satisfactory in some cases. The relatively high cost of the zinc salt and the concentration of the other components of the bath are factors to be considered in selecting the amount. If necessary, the zinc sulfate may be replaced partially with iron sulfate.

The bath may contain other substances ordinarily employed in viscose rayon manufacture. For example, it may contain a very small amount of surface active agents and sodium sulfate. In the present invention it is preferred that the spinbath contain 5 to 20% sodium sulfate. For best results, it has been found that the ratio of the percentage of sodium sulfate to the percentage of zinc sulfate should be between 1.0 and 2.4.

By the use of the low acid and low temperature spinbath of the present invention, either with or without employing a viscose modifier, the stretchability of the freshly spun viscose yarn is increased; and in addition thereto, by adapting the bath temperature to the stretch, it is consequently possible to control the elongation of the yarn as required and still to provide a considerable latitude for bringing the other yarn properties to a most favorable value.

The invention is further illustrated in the following examples:

EXAMPLE I

Alkali cellulose prepared and aged in the usual manner was xanthated with 38% CS_2, based on the cellulose. The xanthate crumbs were dissolved in a solution of sodium hydroxide to produce a viscose having a cellulose content of 6.5% by weight and an alkali content of 5.7% by weight. The viscose was filtered, deaerated and aged for 48 hours to a maturity of 47 gamma number.

This viscose was extruded through a spinneret immersed in a spinbath having the composition by weight of 4.7% H_2SO_4, 10% Na_2SO_4 and 7% $ZnSO_4$. The spinneret employed had 1000 orifices, each having a diameter of 60 microns. The yarn produced thereby had a denier of 1650. The temperature of the spinbath was maintained at 38° C.

The yarn was given a bath travel of 60 cm. after which it was withdrawn by means of a first godet. The yarn was then carried through and stretched in a second bath by means of a second godet and guided therethrough by means of a pair of freely rotatable rollers immersed in the second bath.

The second bath contained 2% H_2SO_4 and had a temperature of 93° C. The yarn withdrawal speed produced by the second godet was 35 meters per minute.

The stretching tension on the yarn as measured between the point at which the yarn is removed from the second bath and the point at which the yarn is taken up by the second godet was maintained at 1500 g. The yarn was aftertreated in the usual way.

For purposes of comparison, yarn was produced in identical manner as described above except the temperature of the spinbath was maintained at 60° C. In each case the yarns were dried without tension. The properties of the yarns together with the stretch imparted therein by the 1500 g. stretching tension are listed in Table I.

Table I

	Spinning Bath Temperature	
	60° C.	38° C.
Stretch percent	100	115
Yarn Properties:		
Dry Strength g./100 den	400	405
Dry Elongation, percent	22	16
Wet Strength, g./100 den	285	300
Wet Elongation, percent	29	23
Degree of Swelling, percent	78	83

It will be noted that the yarn produced with the lower bath temperature had a substantially lower elongation, and an improved strength value as compared with the spinning carried out at 60° C.

Cord made from the yarn using the lower temperature spinbath shows a reduced elongation at break. For example, it is possible by the present invention to reduce the elongation of the cord yarn at a load of 2 to 4 kg.

In order to obtain a dry elongation value of about 11 to 12% which is most desirable for cord rayon yarn, the yarn spun at 38° C. only had to be given an after stretch of 5%. The other yarn to obtain the same elongation had to be given an after stretch of 10%. Also, the shrinkage force, i.e., the force necessary to neutralize the tendency of the yarn to shrink when rewet, was about one-half as much as compared with the second mentioned yarn.

Also, in order to impart a stretch of 100% in the yarn produced at the lower temperature, it was determined that a stretching tension of only 1,000 g. was necessary. This value is 50% less than that necessary in the normal high temperature spinning, which further illustrates the improved results of the present invention.

EXAMPLE II

To illustrate that solely by lowering the spinbath temperature it is possible to lower the elongation without the necessity of giving the yarn additional stretch, the viscose as described in Example I was spun under the same conditions as those given in said example. However, the acid content of the spinbath was 4.4% with the spinbath temperature in this instance being maintained at 30° C. and 39° C. The yarn was stretched 118% between the first and second godet in the second hot acidic bath. Also, the yarn was given an after stretch of 6%. The properties of the yarns obtained under the spinning conditions described herein are listed in Table II.

Table II

	Spinning Bath Temperature	
	30° C.	39° C.
Dry Strength, g./100 den	430	450
Dry Elongation, percent	12.0	14.5
Wet Strength, g./100 den	315	311
Wet Elongation, percent	19	23
Degree of Swelling, percent	81	78

A comparison with the results of the spinning carried

out at 60° C. in Example I shows that employing the lower bath temperature not only reduces elongation but also increases the strength of the yarn. With even lower bath temperatures, it is possible to obtain even lower elongations while maintaining the high strength, as shown by the following example.

EXAMPLE III

Two different viscoses were prepared and each spun in the manner described in Example I into different spinbaths. The viscose and spinbath compositions were so chosen that one process would produce yarn having a relatively low elongation and the second process would produce yarn having a relatively high elongation. Each combination was spun at two different temperatures in order to influence the elongation. The type of yarn with the lower elongation (experiments 1 and 2 in Table III) was spun at 40° C. and 55° C., while the other type (experiments 3 and 4 in table III) was spun at 25° C. and 44° C. and produced substantially the same corresponding elongations. Table III gives the viscose compositions, spinbath compositions, spinbath temperature and the amount of stretch in the hot acidic second bath, together with the properties of the yarn produced therefrom. The yarn was in an oven-dried condition and had not been given a final stretch.

Table III

Experiment No.	1	2	3	4
Viscose Composition:				
Cellulose, Percent	7.3	7.3	6.5	6.5
Alkali, Percent	5.5	5.5	5.8	5.8
Spinbath Composition:				
Percent H_2SO_4	4.5	4.5	5.0	5.0
Percent Na_2SO_4	7.0	7.0	15.0	15.0
Percent $ZnSO_4$	7.0	7.0	5.0	5.0
Spinbath Temp., ° C.	40	55	25	44
Percent Stretch	100	100	120	100
Yarn Properties:				
Kg. Breaking Load	6.9	7.1	6.6	7.2
Percent Elongation	6.2	10.5	5.4	10.4
Breaking Load/Elongation	1.11	0.68	1.22	0.69

It will be noted that in both cases, the lowering of the spinbath temperature results in an increase in the ratio of the strength to the elongation. The present example also illustrates the fact that the temperature range of the present invention is effective when different viscose bath combinations are employed.

EXAMPLE IV

A viscose with a composition of 6.4% cellulose and 5.8% alkali was extruded in the manner described in Example I into a primary spinbath having a temperature of 39° C. and containing 9.5% Na_2SO_4 and 8.5% $ZnSO_4$. The viscose had been ripened for 72 hours to a maturity of 43 gamma number. The spinbath acid content was varied as indicated in Table IV. The freshly spun threads were stretched 114% in the spinbath. The threads were given the three different acid concentrations are given in Table IV.

Table IV

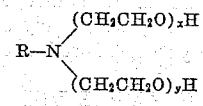
	Acid Concentration of the Spinbath		
	3.8%	4.5%	4.9%
Dry Strength, g./100 den.	434	429	402
Dry Elongation, Percent	16	16	16
Wet Strength, g./100 den.	330	310	298
Wet Elongation, Percent	26	25	23
Degree of Swelling, Percent	72	79	88

It will be noted that the yarn's physical properties are favorably influenced by spinning at low temperatures. For example, with 3.8% H_2SO_4, the swelling is lower and and the strength is higher than when a spinbath temperature of 60° C. is used as in Example I. Also, the cord properties show corresponding improvements.

It has been demonstrated in the foregoing examples that the combination of low acid and low spinbath temperature markedly reduces the elongation and increases the strength of the yarn. The following examples show that the yarn may be further improved by employing a viscose modifier in the spinning operation.

EXAMPLE V

Viscose containing 6.5% cellulose and 5.8% alkali was prepared as in Example I. In this case, however, 0.2% of ethoxylated aliphatic amine of the general formula:



wherein $x+y$ has a mean value of 20, R is a long chain hydrocarbon radical and the average molecular weight thereof is 1,300 was incorporated in the viscose.

The viscose was spun at a gamma number of 48 into a spinbath having the composition of 5% H_2SO_4, 10% Na_2SO_4 and 8% $ZnSO_4$. Spinning was conducted with the temperature of the spinbath being maintained at four different levels. Other conditions such as composition and temperature of the secondary bath, aftertreating of the yarn, and the like, were identical to those of Example I. The yarn properties together with the stretch given the yarn in the secondary bath may be seen by reference to Table V.

Table V

	Spinning Bath Temperatures			
	30° C.	38° C.	45° C.	57° C.
Stretch, Percent	124	120	115	95
Yarn Properties:				
Dry Strength, g./100 den.	480	490	480	490
Elongation Dry, Percent	12.5	14.5	17.0	13.0
Shrinkage, Percent	4.5	4.5	4.5	10
Elongation Dry+Shrinkage, Percent	17	19	21.5	23
Wet Strength, g./100 den.	340	360	360	360
Wet Elongation, Percent	22	24	27	28

It will be noted that the series of tests of this example show that the stretching of yarn can be increased with a lowering of the spinbath temperature. In addition, the sum of the dry elongation plus shrinkage is lower at the lower spinbath temperatures.

EXAMPLE VI

Viscose containing 6.5% cellulose and 5.8% alkali was prepared as in Example I and spun at a gamma number of 48 into a spinbath having the composition of 5% H_2SO_4, 10% Na_2SO_4, 8% $ZnSO_4$ and 0.05% of the ethoxylated aliphatic amine described in Example V. Spinning was conducted while maintaining the temperature of the spinbath at different temperatures, above and within the preferred range of the present invention. The low spinbath temperatures gave much higher yarn strength and reduced elongation than the normal high temperature spinning.

EXAMPLE VII

Viscose containing 6.5% cellulose and 5.8% alkali was prepared as in Example I. 0.2% of the additive employed in Example V was incorporated into the viscose. Spinning was carried out with the spinbath being maintained at temperatures of 30° and 44°. In both cases the yarns were stretched 110% in the secondary bath. The influence of the spinbath temperature on the yarn may be seen by reference to Table VI wherein the physical properties of the yarns are listed.

Table VI

	Spinning Bath Temperatures	
	44° C.	30° C.
Dry Strength, g./100 Den.	440	470
Dry Elongation, Percent	16	12
Wet Strength, g./100 Den.	360	340
Wet Elongation, Percent	31	22
Shrinkage, Percent	6.5	4.8
Elongation Dry+Shrinkage, Percent	22.5	16.8
Secondary Degree of Swelling, Percent	72	67

The stretching tension at the lower spinbath temperature was about 500 g. less. Of course, even better elongation could be obtained by completely utilizing the higher stretchability of the yarn produced at the lower temperature.

EXAMPLE VIII

Viscose containing 7.3% cellulose, 5.0% alkali and 0.2% of the amine additive described in Example V was spun into a spinbath having the composition of 5% H_2SO_4, 10% Na_2SO_4 and 8% $ZnSO_4$. A corresponding relationship between the spinbath temperature on one hand and the stretchability, draw tension and yarn elongation on the other hand was obtained. For example, by using this viscose containing the additive at the low spinbath temperature of the present invention, it was possible to obtain yarn having an air-dry strength of 500 g./100 den. and in some instances even higher.

EXAMPLE IX

Viscose containing 7.3% cellulose, 5.0% alkali and 0.2% of the amine viscose modifier described in Example V was spun into a spinbath having the composition of 5% H_2SO_4, 14% Na_2SO_4 and 6% $ZnSO_4$. A comparison of the yarn properties produced with the spinbath maintained at 57° C. and 46° C. showed that the lower temperature spinning caused a lowering of the cord elongation from 21% to 18% in the air-dry condition and

from 14.5% to 12.5% in the oven-dry condition. Furthermore, at 57° C., the stretching was limited to 100% while at the lower temperature the yarn could be stretched 120% without filamentary breakage. Also, by employing the lower temperature with the attendant higher yarn stretchability, a yarn of greater strength was produced.

Although the invention has been described above in terms of its specific embodiments, certain modifications and equivalents will become apparent to those skilled in the art and are intended to be included within the scope of the present invention, which is to be limited only to the extent of the appended claim.

What is claimed is:

A process for the production of high strength viscose yarn having increased stretchability and low elongation comprising spinning a viscose into a spinbath maintained at a temperature of 38° C. to 39° C. and containing 4 to 6% sulfuric acid, 2 to 15% zinc sulfate and 5 to 20% sodium sulfate, the ratio of the percentage of sodium sulfate to the percentage of zinc sulfate being between 1.0 and 2.4, withdrawing the freshly spun yarn from said spinbath and stretching the yarn in a second hot weakly acidic bath.

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