Feb. 13, 1945. G. I. THURMOND 2,369,191

MANUFACTURE OF RAYON

Original Filed March 1, 1939 4 Sheets-Sheet 1

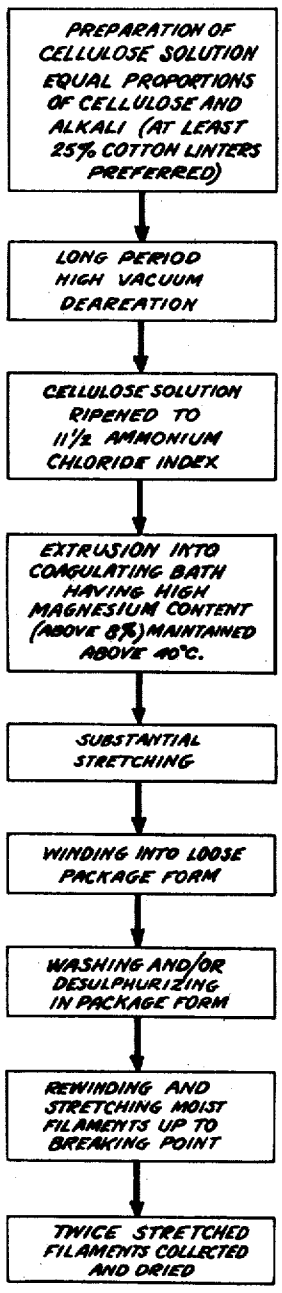


Fig. 1.

Inventor

Gilbert I. Thurmond

By Albin F. Knight

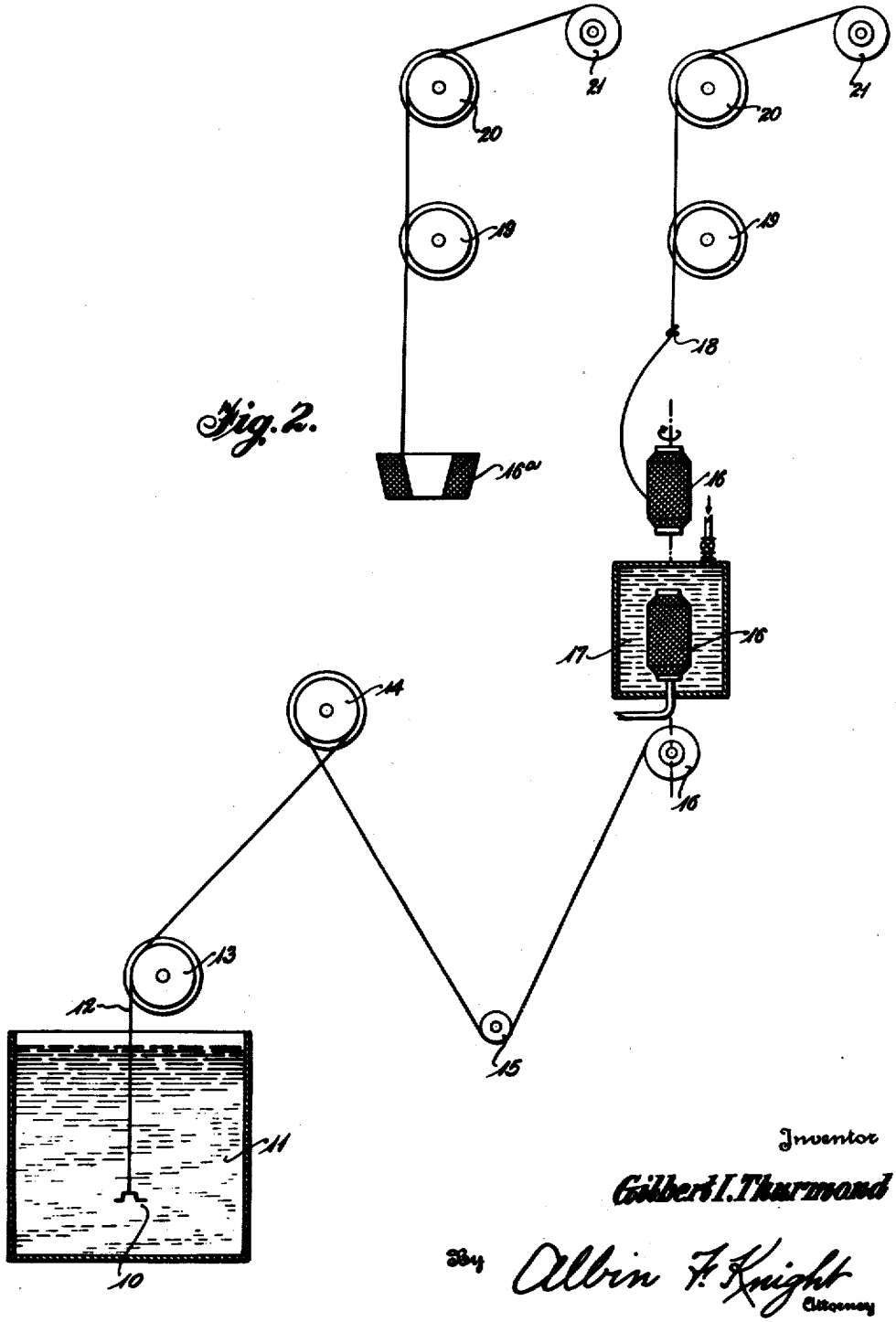
Attorney

Feb. 13, 1945. G. I. THURMOND 2,369,191

MANUFACTURE OF RAYON

Original Filed March 1, 1939 4 Sheets-Sheet 2

Fig. 2.



Inventor

Gilbert I. Thurmond

By Albin F. Knight

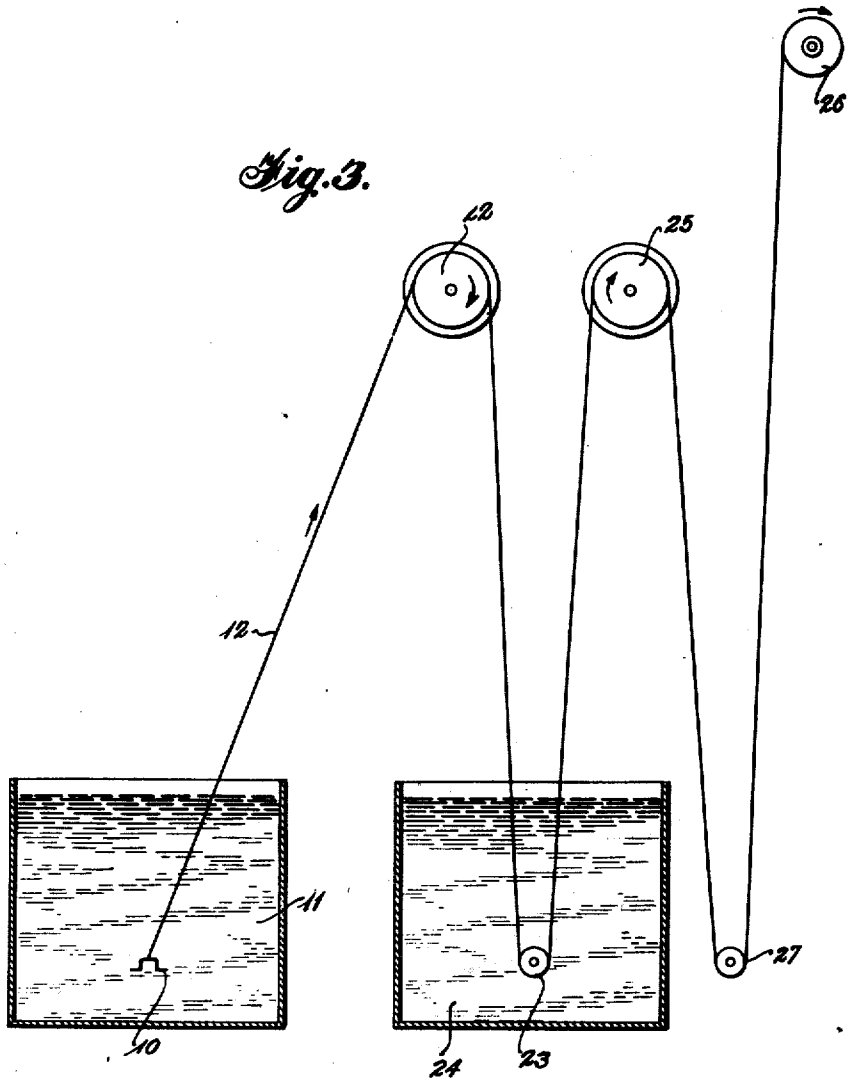
Attorney

Feb. 13, 1945. G. I. THURMOND 2,369,191

MANUFACTURE OF RAYON

Original Filed March 1, 1939 4 Sheets-Sheet 3

Fig. 3.



Inventor

Gilbert I. Thurmond

By Albin F. Knight

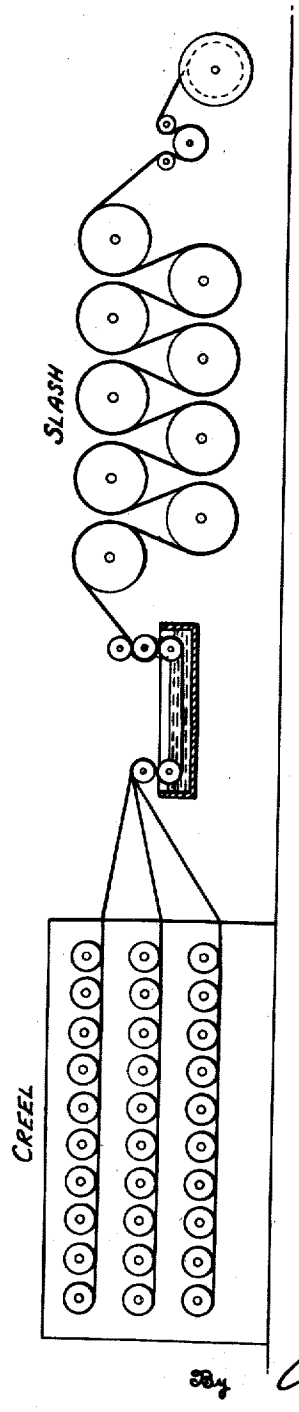
Attorney

Feb. 13, 1945. G. I. THURMOND 2,369,191

MANUFACTURE OF RAYON

Original Filed March 1, 1939 4 Sheets-Sheet 4

Fig. 4.



Inventor
Gilbert I. Thurmond
By Albin F. Knight
Attorney

Patented Feb. 13, 1945

2,369,191

UNITED STATES PATENT OFFICE

2,369,191

MANUFACTURE OF RAYON

Gilbert I. Thurmond, Asheville, N. C., assignor to American Enka Corporation, Enka, N. C., a corporation of Delaware

Original application March 1, 1939, Serial No. 259,261. Divided and this application August 31, 1942, Serial No. 456,805

6 Claims. (Cl. 18—54)

The present application is a division of my application Serial No. 259,261, filed Mar. 1, 1939.

This invention relates to a process of and system for producing a strong yarn or thread of synthetic filaments and to the treatment of yarn or thread in a manner to preserve the properties thereof when formed into a cable of the type normally used in the manufacture of pneumatic tires, hose, belting or reinforcements for molded and cast plastic materials in general of either natural or synthetic origin. More specifically the invention contemplates the formation of improved artificial threads or filaments formed primarily of viscose which have an unusually high tenacity and low elongation.

In the manufacture of pneumatic tires and generally similar flexible rubber articles it has long been the custom to utilize cotton cords for the reinforcement thereof. The substantially universal use of cotton cords in the manufacture of tires has been due primarily to the fact that such cords are not affected by heat and moisture to a prohibitive degree and that when they are formed into strands twisted in a definite manner they have the required degree of resiliency, flexibility and tenacity. However, during recent years many tests have shown that cords formed of rayon may be utilized in tires and like construction with even greater effectiveness than cords produced from cotton. Nevertheless, the substitution of rayon cords or cables for cotton cords in the manufacture of products of the type indicated has been delayed for numerous difficulties were encountered which had to be more fully overcome before rayon cords were suitable for extensive commercial adoption.

In view of the research and other activities of the manufacturers of tires, belts and allied products there is at present a demand for high quality rayon threads and yarns at a cost somewhat comparable with cotton, and this demand will undoubtedly increase rapidly as yarns are produced which have a greater strength and lower elongation and when cables formed therefrom have higher flexing tests and adhere satisfactorily to rubber. Customarily tire cords are formed in three separate twisting operations, the basic threads which are termed yarn comprise a plurality of small filaments twisted together. A plurality of yarns are twisted together or "plied" to form a structure known as a ply or intermediate strand and plies thus formed are twisted with other plies to form a cord or cable. It has been determined that the properties of the cable are dependent in part upon the degree of twist at each stage in the formation thereof. Preferably the yarn has a very low degree of twist.

In connection with the use of rayon in the production of cables for use as reinforcements in tires, belts and the like, the properties of the individual filaments and of the slightly twisted yarn determine to a substantial degree the final properties of the cables or cords. While the strength per unit weight and elongation of the cable or cord may be markedly impaired by improper twisting at its various stages of formation yet the ratio of corresponding values for filaments and cords will be very nearly constant for a given method of twisting.

In the manufacture of pneumatic tires, cords are impregnated with and covered by rubber which is then vulcanized and the strength of the tire walls is dependent to a large extent upon the properties of the cord imbedded therein. It is clear that a strong cord is required for use in tires. However, disadvantages other than those naturally resulting from weakness of the fiber arise from the use of inferior cords even when formed of high quality fibers and the reasons therefor are not so readily apparent. If there is any tendency on the part of the cord to alter its length due to its original inferior qualities or because of deterioration or aging, the bond between adjacent cords and between the cord and rubber may be ruptured to such extent as to render the tires practically useless. Nevertheless, the cord must have a substantial resiliency in order to withstand the strains normally imposed by reason of the pneumatic pressure within and of mechanical shocks applied to the exterior of the tire casing. Rayon threads and cables known prior to this invention do not possess a suitable combination of high tenacity, resilience and low degree of elongation to adapt them for use in bodies formed from plastic compositions, such as tires, belts, and the like.

This invention contemplates the provision of a superior type of artificial filaments and yarn formed thereof, which overcomes the difficulties referred to above and which have a high tenacity and other properties very desirable in the production of cords for the fabrication of pneumatic tires and the like. It has as a further object the provision of a process for the formation of filaments and yarns of uniform properties.

Additionally the invention contemplates a process of and a system for producing a strong and extremely low elongation artificial yarn, wherein the freshly formed filaments are stretched, for example, up to the breaking point,

washed, and thereafter in the acid free condition they are stretched again and as a result of which treatment a greater stretching of the filaments is obtained than has heretofore been considered possible.

It is also an important object of this invention to provide a rayon cord or cable having increased resilience and very little tendency to change in length as a result of alteration in the properties thereof.

Other objects and advantages of the present invention will be apparent from the following detailed description when considered in connection with the accompanying drawings wherein:

Figure 1 is a flow sheet setting forth the important steps of one complete interrupted process conducted in accordance with the invention;

Figure 2 is a diagrammatic view of an apparatus which includes a twisting device used in one step of the alternative interrupted process of Figure 1;

Figure 3 is another modification shown diagrammatically of the invention wherein the major stretching operation takes place between driven rollers and during washing; and

Figure 4 is a schematic view of a plurality of packages being unwound from a creel, passed through a slasher and collected on a beam.

In referring to the drawings in detail with particular reference to Figure 1, it will be seen that the present invention when applied to the production of viscose yarn comprises generally the preparation of a viscose spinning solution of alkali and cellulose, the latter preferably comprising at least 25% of cotton linters. The solution is then aged, filtered and thoroughly deaerated. Such deaeration of the viscose is usually accomplished under vacuum, in which event the deaeration step is preferably practiced by subjecting the viscose solution to pressures abnormally low as compared with known technique for an abnormally prolonged period of time. For example, a vacuum below 20 mm. may be desirable. The spinning solution thus treated is then delivered to spinnerets through corrosion resistant and non-contaminating conduits, spun into a coagulating bath and the yarn composed of a number of filaments is thereafter stretched a plurality of times. In the spinning arrangement shown in Figure 3, the yarn is only given a slight stretch between the spinneret and first godet, but by virtue of the washing action in the second bath which is only slightly acidic, a very much greater stretch can be obtained between the two godets than has heretofore been possible. When the yarn is stretched initially up to its breaking point while in acid condition and then is washed and again stretched up to its breaking point while in a moist and substantially acid-free condition it will have a very high tensile strength, and a low degree of elongation. However, filaments which are stretched to a lesser degree during etiher of the stretching steps or both will nevertheless be suited for use in tires and they will have substantially uniform properties throughout their lengths.

It has also been determined that very efficient results may be had if the filaments are stretched but once providing a high degree of stretch is imparted thereto. This is particularly true if the stretching step is conducted during, or subsequent to, the partial washing of the filaments and while they are still in a plastic state.

More specifically, as will appear from a consideration of Figure 1 of the drawings, very nigh quality filaments are obtained by the utilization of a viscose solution which contains about 7.4% cellulose by weight and an equal amount of sodium hydroxide. Although the viscose solution employed according to this invention is characterized by a content of cellulose substantially equal to the amount of caustic employed therein, the proportions may be varied within rather wide limits with resultant loss of but some of the benefits of the invention. After the preparation of the viscose solution it is subjected to high vacuum deaeration for a prolonged period. The deaerated solution is then aged or ripened. Preferably it is ripened to or above 11½ ammonium chloride index. During the aging period the viscose solution is filtered in a filtration apparatus composed of materials resistant to attack by components of the solution. A filter press may be utilized for this purpose, the frames of the press and other parts which come in contact with the solution being formed of, or covered with, nickel or other suitable material which will not contaminate the solution undergoing treatment. After the viscose solution has been properly ripened and it has been filtered it is passed through conduits similarly formed or lined with non-contaminating material and it is delivered to spinnerets through which it is extruded into a spinning or coagulating bath.

The filaments thus formed are then stretched and further treated to provide a yarn having a high tensile strength, a low degree of elongation particularly suited to the formation of tire cords and one which has substantially uniform properties throughout its length.

In general the spinning bath is of somewhat conventional nature except with respect to the magnesium content and as hereinafter specified. Other components dissolved therein may include sulphuric acid, sodium sulphate, ammonium sulphate, zinc sulphate and other salts when desirable. Such baths, as are known in the art, give considerable difficulty during the spinning operation by reason of the fact that when they are used solids tend to accumulate in the spinneret orifices and impair the properties of the filaments. In order to produce a yarn satisfactory for general use frequent cleaning of the spinnerets was found to be necessary and this cleaning operation of the small orifices necessarily involves costly precision work. However, this problem may now be substantially entirely overcome and a great improvement in the properties of the filaments produced will be had by spinning the filaments in the presence of a dissolved material of a cation-active nature. Such materials are discussed at length in United States Patent 2,125,031 to Polak, et al. For example, a small quantity (0.04 per cent by weight) of dodecyl pyridinium chloride dissolved in the spinning bath eliminates practically all of the irregularities and difficulties of spinning and permits a much longer spinning time free from the frequent interruptions heretofore encountered for cleaning the spinneret.

The contamination of the spinnerets to an exaggerated degree has ben encountered when an ordinary viscose solution is extruded through gold-palladium spinnerets into a spinning bath of the type noted above. The spinnerets become seriously contaminated in a few hours and this incurs interruption of the spinning period with resultant time and production losses. However, the presence of 0.1% of dodecyl triethyl ammo-

nium iodide in the spinning solution reduces the tendency toward clogging of the spinneret orifices to a remarkable extent.

The filaments formed by the extrusion step are conducted through the bath and upon being withdrawn therefrom they are given a suitable stretch, such as for example from about 30% up to the breaking point. The filaments are then again stretched while in moist condition from about 10% up to the breaking point. Between the two stretching steps the filaments are preferably washed and desulphurized or at least they are partially washed since the intermediate washing step causes the filaments to be stronger and to have a lower elongation than when the step is omitted. It is surprising to note that by following such a procedure considerably more stretch can be imparted to the filaments than has heretofore been possible. A total stretch can be obtained that is substantially above 50% and it has even been determined that the filaments can be stretched more than 70% of their original length without breaking. Washing and desulphurizing of the filaments may be accomplished in several ways, one being shown in the flow sheet (Figure 1) of the drawings wherein the coagulated filaments are stretched and then wound upon a spool under reduced tension, whereupon the bobbin is washed by the pressure-vacuum method and the moist filaments are then either twisted and stretched during a rewinding operation or they are merely stretched and not twisted. Cake spun yarn having been twisted during collection has to be stretched only.

The interrupted process permits the winding of a spool of freshly spun yarn under less tension than heretofore utilized and this facilitates easy washing of the yarn while in spool form. During the rewinding the yarn is wound in the usual manner. Ease of washing is an important feature of this phase of the invention. In this connection it should be noted that while the second stretching step may be performed before complete washing, such procedure then involves the washing of a tightly wound bobbin, but this is permissible because the package contains only a small amount of residual acid from the precipitating bath.

In referring to Figures 2 and 3 inclusive, the systems illustrated are constructed in accordance with specific features of this invention. Yarns having the characteristics suitable for use in the manufacture of tires and the like may be readily produced with either of the systems by the proper preparation and handling of the spinning solution in the bath and by the control of the stretching operation. While artificial yarns other than viscose yarns may be produced by the methods herein disclosed with satisfactory results, it will suffice to refer to the apparatus illustrated when used in connection with the viscose process.

In the system shown in Figure 2, a spinneret 10 is connected to a feed pipe (not shown) through which a viscose solution properly prepared, deaerated and ripened, is delivered thereto. The solution is then extruded through the spinneret into a coagulating bath 11 of the type referred to above, and the yarn 12 formed of a number of filaments is drawn from the bath by a godet wheel 13. The yarn is propelled by the godet 13 at the same speed as the peripheral speed thereof and the yarn is led one or more times therearound to avoid slippage upon its surface. Another godet wheel 14 is arranged in spaced relation to godet wheel 13 and it is rotated at a greater peripheral speed than is the first godet wheel, whereby, when the yarn is passed from the godet wheel 13 and is directed over and around godet wheel 14 one or more times, it will of necessity be stretched between the two godet wheels, the desired degree of stretching being controlled by the relative speeds of the godet wheels.

The filaments are then passed from godet wheel 14 to and around idler 15, whereupon they are passed to the temporary bobbin 16 where they are collected under reduced tension. It will be noted in the system as thus far described only one stretching is imparted to the filaments up to the time of collection on bobbin 16. Bobbin 16 is next subjected to a high-pressure, high-vacuum washing and/or other treating operation in a bath 17. Bobbin 16, after being treated, and while the filaments are still in a plastic condition, is placed upon a stationary support and the filaments are drawn therefrom and twisted. The filaments pass through guide 18 and then around godet wheel 19 one or more times, and they are then passed to and around godet wheel 20, after which they are delivered to bobbin 21 for collection in the usual manner. Godet wheels 19 and 20 are substantially the same as godet wheels 13 and 14 and the filaments are conducted thereover in a similar manner. In order to effect a second stretching of the filaments as they are passed between godet wheels 19 and 20, godet wheel 20 is driven in such a manner that its peripheral speed is increased to the desired extent above the peripheral speed of godet wheel 19. In the event that the freshly spun filaments are collected in a centrifugal bucket, the cake is made up of already twisted filaments, in which case it is only necessary to restretch and collect the filaments in a manner also shown in Figure 2. It is, of course, within the scope of the invention to dry the washed bobbin or cake spun package prior to the re-stretching operation. The dried package must then be rewetted before or during the second stretch. The washed bobbin package 16 may even be satisfactorily restretched without twisting, the twisting step being conducted in a subsequent separate operation. When the package 16 or 16a is restretched between the godets 19 and 20, a certain amount of drying will result, especially if the twisting is simultaneously effected. If it is desired to completely dry the packages either in bobbin or cake form, a drying zone can be readily interposed between the last godet 20 and the take-up device 21. It has been determined that such treatment will result in yarn having extremely uniform shrinkage properties throughout its length.

Referring now to Figure 3, the filaments 12 are extruded through spinneret 10 into a coagulating bath 11 and thence led one or more times around a godet wheel 22 whereupon the filaments are conducted to an idler roller 23 located in a wash bath 24. This bath may be very slightly acidic in order to further coagulate the filaments while at the same time effect a substantial washing thereof. After the filaments leave the wash bath 24, they are wrapped one or more times around a godet wheel 25 rotating at a higher peripheral speed than godet wheel 22. This difference in peripheral speed imparts the desired stretch to the filaments which taken in conjunction with the initial stretch in bath 11, can be considerably greater than was heretofore possible by conventional methods. The filaments

are then collected on a bobbin 26 or in a pot after first passing around a second idler roller 27.

It is apparent from the foregoing that in the present process in which a high stretch is only imparted at one stage, the threads may be initially collected under the desired tension in acid condition, either partially washed or unwashed. After the complete washing and other aftertreatments have been effected, the package is rewound at which time a multiplicity of such threads are given an additional stretch and dried on a slasher and finally collected at a common source such as a beam or the like (see Figure 4). Of course, if the initial package is washed and dried in package form, it must be rewetted prior to restretching and drying on the slasher.

The yarn produced by the utilization of either of the above systems in connection with the process of this invention has, as stated above, important application to the tire and related arts. An important step in the preparation of a tire cord for the purpose of preventing loss of strength due to the twisting of the cord components involves the application of a finishing material to the yarn in at least one stage of the spinning operation. A softening agent which has no deteriorating effect on the yarn is utilized as the finishing material. This is a highly important aspect of the complete process of this invention since it has been found that the employment of the finishing material will improve the final strength of the cable by from 10 to 50% by preventing substantial loss in strength of the yarn during twisting. Apparently, the employment of the finishing material permits the yarns and plies when twisted to assume a position of least tension and strain and specific flexing tests have been made on cables formed of yarn twisted as hereinafter specified which show abnormally high flexing tests as compared to yarn otherwise similar but not finished in the same manner. Additionally, cables formed from yarns finished in this manner are smoother and more dense than similar cables formed of untreated yarns and the strength thereof closely approaches the combined strength of the individual yarns. A solution of Monopole Brilliant oil has been found to produce excellent results when used as a finish for the yarn. However, various types of materials can be advantageously used for this purpose and sulphonated olive oil, sulphonated castor oil, emulsified glycerides such as blown tea-seed oil, and lecithin have been found satisfactory. Sulphonated oil for this purpose may be stabilized by the addition thereto of a small amount of alkali such as soap and an anti-oxidant.

The finishing agent may be applied to the yarn as such or as a solution or emulsion with any suitable vehicle. Good effects are obtained from the addition of a finish during washing or immediately subsequent to the washing stage. However, application of the treating material to the dried spools of yarn with redrying prior to the twisting of the yarn gives excellent results. It has been found that 0.3% of monopole oil based on the weight of the dry yarn will provide one of the best concentrations for most purposes. Results which have been obtained indicate very clearly that softening agents are much better than lubricating finishes in connection with yarns to be plied or plied and cabled and then used in the production of cord, tires and the like. For example, a straight mineral oil finish has little or no effect in conserving the strength of the yarn to give a strong cable, while a softening agent yields improvements in strength running as high as 50% over cables formed from yarns not so finished. It would seem that such finishing of the yarn is essential for the production of a smooth, well-formed, compact cable which will have the maximum strength obtainable from a plurality of twisted yarns.

It is of course desirable that the yarn be lubricated to some extent to prevent breakage or other damage during handling and if the softening agent does not impart sufficient lubrication thereto, a suitable lubricant may be blended with the softening agent.

Tire cords and reinforcing cables and fabrics used in various bodies formed of plastic material may be prepared from rayon in the manner noted above with numerous not heretofore obtainable advantages. In addition to the points of superiority already noted, the tendency of rayon cord in tires to "grow" in service is prevented or minimized by the use of the yarns of low elongation described above. It will be readily seen that the tire structure will be adversely affected or the tire per se enlarges if the yarns used in producing the reinforcing cables becomes permanently elongated during use of the tire.

To better set forth a comparison of the improvements of the various features of this invention the details of one specific process for producing a rayon cable will be described without the additional stretch but with the conditioning operation and other novel features merely by way of exemplification. A soaking lye is first prepared from 18.3% alkali containing 0.6% or 0.7% hemicellulose. Alkali cellulose is next prepared and at least 25% of cotton linters is utilized with the remainder of the cellulose being derived from wood pulp. The alkali cellulose is shredded in a normal manner for two hours and is subjected to an ageing temperature of 21° C. for approximately 56 hours. Viscose having a cellulose content of 7.4% and alkali content of 7.3% is next formed therefrom, and it is permitted to mature or ripen to 12.3 ammonium chloride index whereupon the viscosity is approximately 40 seconds. The viscose is then extruded through a spinneret into a coagulating bath containing sulphuric acid, sodium sulphate, zinc sulphate and approximately 10% of magnesium sulphate. The spinning bath is maintained at a temperature of 43° C. to 45° C., and a higher temperature may be utilized if proper equipment is available to avoid evaporation problems encountered with the likely impairment to the health of the operators. The filaments thus formed are conducted from the bath and have a stretch imparted thereto of 45% by passing the same between two godets, and thence to a spinning spool that has a takeup speed substantially equal to the peripheral speed of the second godet. This corresponds to the first part of the interrupted process. The spinning speed utilized is 71 meters per minute, the yarn being 275 deniers and having 120 filaments. Yarn thus produced is washed acid free and then dried and it is finished by being further dried by the vacuum method. A solution of 0.6% Monopole Brilliant oil is caused to permeate the wound package and the package is subjected to dry vacuum to insure uniform distribution of the finish. The package is then additionally dried on the spinning spool. After being dried, the yarn is then twisted four turns per inch. The yarn in this condition is ready for formation into cables and in tests which have been conducted to determine the

flexibility of cables formed from such yarn, five of the thus formed yarns were twisted together to form a ply or intermediate strand and three such strands were then twisted together to form a cable. The flexing or fatigue test of such a cable was 35,235 flexes before breaking and a cable similarly produced but in which the viscose was formed entirely from wood pulp without the inclusion of cotton linters and the coagulating bath did not have a high magnesium content, a flexing test of 24,975 was determined. The same flexing test made with respect to a cable formed of yarns which were not finished with Monopole Brilliant oil or any of the other materials referred to above, had a flexing test of only about 900. The fatigue or flexing tests were conducted on a similar principle as that taught in an article by F. B. W. King and R. Truesdale, published in The Textile Recorder, May 15, 1923.

The other physical characteristics of the yarn are tabulated below in order to show by way of comparison the improved results obtained:

	Yarn				Cable	
	Dry strength	Wet strength	Dry elongation	Wet elongation	Strength	Elongation
			Per cent	*Per cent*		*Per cent*
Prepared from normal viscose	281	162	11	14	13	14
Prepared from normal viscose + softening agent	281	162	11	14	18	9.4
Prepared according to above example	318	185	12	15	19.9	9
Prepared according to above example + additional 15% stretch	340	207	6	12.5	23	6.5

The strength of the yarn is given in grams per 100 denier and the strength of the cable is given in pounds; in addition, the strength of both the yarn and cable was obtained by conditioning at 65% relative humidity.

Yarn prepared in accordance with the above example but collected in a centrifugal bucket does not have as much strength or as low elongation as yarn collected on a bobbin. This is due to lack of tension present in the centrifugal bucket process. However, an additional stretch will cause the yarn to compare favorably with yarn collected on a bobbin. The following table indicates the properties of such yarn:

	Dry strength	Wet strength	Dry elongation	Wet elongation
Before 2nd stretch	286	159	13	20
5–10% 2nd stretch	310	173	8	18
10–20% 2nd stretch	332	186	6	15

The importance of stretching after washing and subsequent proper finishing to increase the cable strength, to prevent loss of strength during cabling, and to obtain a cable of low elongation is obvious from the preceding examples.

In the utilization of this invention, it is extremely important that all apparatus, used in the preparation of the yarns, that contact with the substances entering into the formation thereof, be composed of a material which resists corrosion and which will not contaminate said substances. It has long been known that porcelain, nickel and the like and stainless steel are suitable materials for use in constructing the filter pressesr, conduits, and the like or as linings or coating therefor.

It is to be understood that while the discussion is generally directed to the preparation of filaments, it is intended to cover threads, yarns, cords, ribbons, foils, sheets or other products usually produced by extrusion or spinning. The invention has been described in detail in order that those skilled in the art may practice the same, but it is obvious that it is not to be restricted to the specific examples set forth.

In using the expression "up to the breaking point" throughout the specification and claims, it is, of course, intended to mean that the thread is stretched just short of the breaking point.

What is claimed is:

1. A method of preparing freshly spun viscose rayon suitable for use in reinforcing rubber articles and the like which comprises the steps of extruding a viscose solution into a coagulating bath to form a thread consisting of a multiplicity of filaments, withdrawing the thread from the bath and positively stretching the same while it is in at least a slightly acid condition and during partial washing, temporarily collecting the thread in package form under reduced tension, after-treating the thread in package form including the steps of washing and drying, unwinding, rewetting, and then simultaneously restretching and redrying the thread whereby the cumulative stretching steps provide a high tenacity thread and the drying under tension effects a low elongation thereof.

2. A method of preparing freshly spun viscose rayon suitable for use in reinforcing rubber articles and the like which comprises the steps of extruding a viscose solution into a coagulating bath to form a thread consisting of a multiplicity of filaments, withdrawing the thread from the bath and positively stretching the same in acid condition and during partial washing, temporarily collecting the thread in package form under reduced tension, washing the said package substantially acid-free, drawing the thread from the package and drying the same under sufficient tension to again stretch the thread whereby the cumulative stretching steps provide a high tenacity thread and the drying under tension effects a low elongation thereof.

3. A method of preparing freshly spun viscose rayon suitable for use in reinforcing rubber articles and the like which comprises the steps of extruding a viscose solution into a coagulating bath to form a thread consisting of a multiplicity of filaments, withdrawing the thread from the bath and positively stretching the same during partial washing, temporarily collecting the thread in package form, completing the washing of said package, unwinding, rewetting, and then redrying the thread while restretching the same and drying the same whereby the cumulative stretching steps provide a high tenacity thread and the drying under tension effects a low elongation thereof.

4. A method of preparing freshly spun viscose rayon suitable for use in reinforcing rubber articles and the like which comprises the steps

of extruding a viscose solution into a coagulating bath to form a thread consisting of a multiplicity of filaments, withdrawing the thread from the bath and positively stretching the same during partial washing above 50%, temporarily collecting the thread in package form, completing the washing thereof, drawing the thread from the package and finally, simultaneously restretching and drying whereby the cumulative stretching steps provide a high tenacity thread and the drying under tension effects a low elongation thereof.

5. A process of preparing products containing thread of high tenacity and low elongation comprising preparing a viscose spinning solution, the cellulose content of which is derived from at least 25% cotton linters, extruding the viscose solution into an acid coagulating bath containing metallic sulphates to form a thread consisting of a multiplicity of filaments, drawing the thread from the coagulating bath and positively stretching the same during partial washing substantially above 50%, collecting the stretched thread in package form, washing a plurality of such packages substantially acid-free, finishing and drying the thread while effecting an additional stretch on a slasher to reduce the elongation thereof and finally collecting same on a beam.

6. A process of preparing products containing thread of high tenacity and low elongation comprising preparing a viscose spinning solution, the cellulose content of which is derived from at least 25% cotton linters, extruding the viscose solution into an acid coagulating bath containing metallic sulphates to form a thread consisting of a multiplicity of filaments, drawing the thread from the coagulating bath and positively stretching the same during partial washing substantially above 50%, collecting the stretched thread in package form under reduced tension, washing the said packages substantially acid-free and drying the same, unwinding a plurality of such packages and rewetting and redrying the thread while effecting an additional stretch on a slasher to reduce the elongation thereof and finally collecting the thread, being passed from the slasher, on a beam.

GILBERT I. THURMOND.

CERTIFICATE OF CORRECTION.

Patent No. 2,369,191. February 13, 1945.

GILBERT I. THURMOND.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 5, second column, lines 68 and 69, claim 3, strike out the words "and drying the same" and insert the same after "package" and before the comma in line 67, same claim; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 15th day of May, A. D. 1945.

Leslie Frazer

(Seal) Acting Commissioner of Patents.

of extruding a viscose solution into a coagulating bath to form a thread consisting of a multiplicity of filaments, withdrawing the thread from the bath and positively stretching the same during partial washing above 50%, temporarily collecting the thread in package form, completing the washing thereof, drawing the thread from the package and finally, simultaneously restretching and drying whereby the cumulative stretching steps provide a high tenacity thread and the drying under tension effects a low elongation thereof.

5. A process of preparing products containing thread of high tenacity and low elongation comprising preparing a viscose spinning solution, the cellulose content of which is derived from at least 25% cotton linters, extruding the viscose solution into an acid coagulating bath containing metallic sulphates to form a thread consisting of a multiplicity of filaments, drawing the thread from the coagulating bath and positively stretching the same during partial washing substantially above 50%, collecting the stretched thread in package form, washing a plurality of such packages substantially acid-free, finishing and drying the thread while effecting an additional stretch on a slasher to reduce the elongation thereof and finally collecting same on a beam.

6. A process of preparing products containing thread of high tenacity and low elongation comprising preparing a viscose spinning solution, the cellulose content of which is derived from at least 25% cotton linters, extruding the viscose solution into an acid coagulating bath containing metallic sulphates to form a thread consisting of a multiplicity of filaments, drawing the thread from the coagulating bath and positively stretching the same during partial washing substantially above 50%, collecting the stretched thread in package form under reduced tension, washing the said packages substantially acid-free and drying the same, unwinding a plurality of such packages and rewetting and redrying the thread while effecting an additional stretch on a slasher to reduce the elongation thereof and finally collecting the thread, being passed from the slasher, on a beam.

GILBERT I. THURMOND.

CERTIFICATE OF CORRECTION.

Patent No. 2,369,191. February 13, 1945.

GILBERT I. THURMOND.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 5, second column, lines 68 and 69, claim 3, strike out the words "and drying the same" and insert the same after "package" and before the comma in line 67, same claim; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 15th day of May, A. D. 1945.

Leslie Frazer

(Seal) Acting Commissioner of Patents.