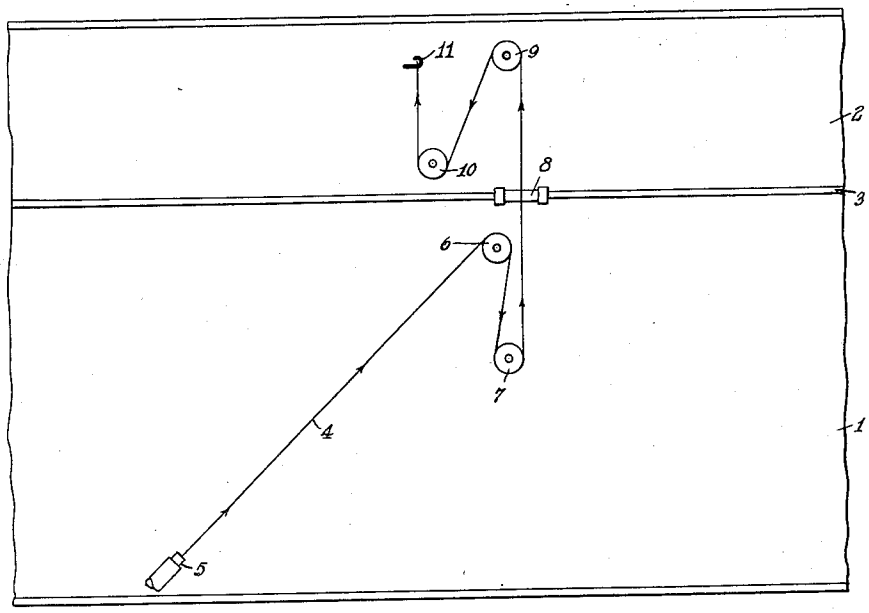
Oct. 18, 1938. H. H. PARKER 2,133,715

METHOD OF PRODUCING ARTIFICIAL THREAD

Original Filed Aug. 23, 1933

Fig. 1.



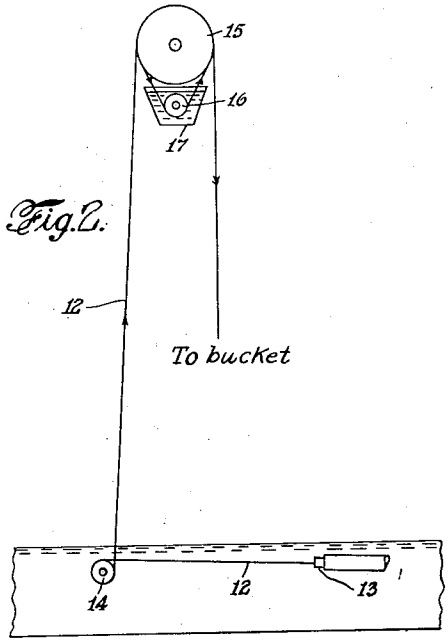


Fig. 2.

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METHOD OF PRODUCING ARTIFICIAL THREAD

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Application August 23, 1933, Serial No. 686,353
Renewed March 16, 1938

6 Claims. (Cl. 18—54)

This invention relates to the manufacture of high tenacity yarn, thread, filaments, straw, ribbon or the like.

Recently, there has been proposed a method of producing a new type of regenerated cellulose threads possessing a very high dry tenacity, i. e. in excess of 2 grams per denier, by spinning viscose into a bath containing more than 50% sulphuric acid. According to this procedure, tension is applied to the freshly formed thread by passing it between a series of glass rods so placed that the thread must bend therearound in its travel. Owing to the great strain imposed upon the weak and tender structure of the plasticized thread by the frictional effect of the glass rods, the speed of spinning is relatively low and many of the filaments become damaged and broken, so that the ultimate product is seldom, if ever, free from undue broken filaments and is not only poor in quality but greatly reduced in strength. For this reason alone, such a process is of doubtful commercial value. Furthermore, when tension is applied by means of sliding friction, it is necessary to spin at very low speeds, which is also a serious drawback to commercial success. Also, the yarn produced by such a procedure possesses undesirable non-uniformity of physical and chemical properties which are present in all yarns subjected in spinning to an irregular stretch, because the spinning tension must necessarily vary due to the impossibility of keeping the spinning conditions exactly the same at all times.

I have found that I can overcome the above-mentioned disadvantages by spinning a thread in a spinning bath containing a plasticizing ingredient, such as a bath containing from at least 45%–85% or more sulphuric acid, and applying a substantially uniform tension, preferably in stages, to the coagulated and partially regenerated thread without subjecting said thread to severe sliding friction, such as that developed when a thread is dragged under tension around a rod or other guide, and produce a strong thread of higher quality than that which has been heretofore produced as well as spin at a much higher speed than has been considered possible up to the present time.

It is, therefore, an object of this invention to provide a method of spinning a thread in a plasticizing bath and applying a substantially uniform tension, preferably in stages, to the thread without subjecting said thread to severe sliding friction, such as that developed when a thread is dragged under tension around a rod or other guide, thereby producing a uniform thread of improved strength and elongation which will dye more uniformly and which is substantially free from broken filaments.

Another object of my invention is to provide a method for orderly and commercially spinning at high speeds high tenacity threads of regenerated cellulose in a plasticizing bath.

A still further object of my invention is to produce a high quality thread of good strength, elasticity and endurance which is especially suited for such uses as the manufacture of cords or fabrics for use in vehicle tires, steam hose, of reinforcing fabrics, brake linings, etc.

Other objects will become apparent from the following description, appended claims and accompanying drawing in which:

Figure 1 is a plan view of an illustrative diagrammatic set-up for carrying out one embodiment of the instant invention; and

Figure 2 is a front elevation of an illustrative diagrammatic set-up for carrying out another embodiment of the instant invention.

In accordance with the principles of this invention, I spin a viscose solution into a plasticizing bath, such as one containing 45%–85% of sulphuric acid, and impose on the thread, at one or more points in its path of travel from the spinneret to the draw-off device, a tension which is substantial, uniform, constant and substantially free from any undue or substantial sliding friction, such as that developed when a thread is dragged under tension under a rod or other guide. After passage through the acid bath, further action of the acid is checked, such as by passing the thread through a water bath.

As illustrative means for imposing the substantial, uniform and constant tension, I use rotatably mounted rollers which are rotated solely by thread passing therearound and provided with means for developing a resistance to rotation in the bath, such as those described in British Patent No. 318,258 and in United States Patent No. 1,878,455. In the preferred form of the invention, I utilize a plurality of the guides arranged in series and with or without other types of guides, such as those of the friction type. For example, two or three floating guide rollers, such as are shown and described in United States Patent No. 1,878,455, may be used alone and/or in combination with one or more rotating guides, such as is described in the British patent supra.

The rotating guides above mentioned are so devised and constructed as to compensate, as explained in application Serial No. 655,738, for the varying differences in a viscose concentration of

bath, temperature, etc., that always exist in the commercial manufacture of regenerated cellulose threads by speeding up or slowing down, in accordance with the ease or difficulty of tensioning the freshly formed thread.

In the preferred form of this invention, I employ a viscose solution of the type specifically described in my copending application Serial No. 676,463. This viscose solution may be prepared by using such materials, apparatus and procedures as will not cause the resulting viscose to be contaminated with the undesirable impurities. According to one procedure, the viscose, after ripening, is conducted to the extrusion device through equipment made of a material which will not be corroded by the viscose, will not contaminate the viscose and will not promote the gelation of the viscose. The viscose which is preferred in the instant invention may also be of the ordinary type which, after considerable filtration such as through a nickel filter press, is delivered to the extrusion device without change in quality, such as by being conducted, after leaving the nickel filter press, through equipment made of any material such as will not be corroded by the viscose, will not contaminate the viscose and will not promote the gelation of the viscose. Although I prefer nickel equipment, I can conveniently use equipment made of materials such as glass, rubber, alkali-resisting molded compositions or the commercial alloys known as "Illium", either alone or in combination. The preferred viscose, which is delivered to the extrusion devices without change in quality, enables me to spin in a more orderly manner and at higher tensions which are substantially constant at all times. Furthermore, due to the fact that I apply substantially uniform tension, I am able to spin high quality thread at very high speeds, namely, 3000 or more inches per minute.

Any suitable viscose solution may be used in the present process. Satisfactory results are secured with viscoses prepared from aged alkali cellulose and xanthated with 30 to 60 or more parts by weight based on the cellulose. The preferred viscose of this type contains 7% cellulose, 6% alkali and has a salt index of 8. Satisfactory results are also obtained by utilizing viscose solutions prepared from cellulose xanthated with not more than 25 parts by weight based on the cellulose, such as set forth in my copending application Serial No. 501,473.

The spinning bath contemplated by the instant invention contains a concentration of sulphuric acid which will exert a plasticizing effect on the freshly produced thread. In general, a spinning bath containing at least 45% of acid will produce satisfactory results, although baths containing up to 85%, or indeed more, may also be used.

The total bath travel of the thread in the acid bath will vary within wide limits, depending partly on the size of the thread being produced. For example, in making a 100 denier-40 filament thread, a bath travel of from 10 to 40 inches has given satisfactory results. In methods or processes producing threads of larger denier and larger filaments, the bath travel through the acid bath will be as much as 60 inches or indeed more. On the other hand, with thread of fine denier and fine filaments, such as 100 denier-100 filaments, the bath travel should not exceed 15 to 20 inches and may even be as small as 5 inches.

The travel of the thread in the water bath is of such distance as may be necessary to check the action of the acid.

The total tension which may be developed in the thread may vary from 0.1 to 0.6 gram per denier or higher. In the preferred embodiment, the total tension in the thread is developed progressively and in stages. It is to be noted that by "total tension" I mean the tension on the thread between the last tension-applying device in the bath and the traverse guide (in the case of bobbin spinning) or (in the case of bucket spinning) the feed wheel.

The instant invention contemplates spinning a thread at a high speed, such as not less than 3,000 inches per minute and indeed 3,600 inches per minute or higher.

The following examples are given to further illustrate my invention, although I do not intend to be limited thereby:

Example 1.—100 parts of a high grade wood pulp, such as is commonly used in the preparation of viscose, and containing 9% to 10% moisture, are steeped in caustic soda of 17% to 20% strength at about 18° C. for 1 to 3 hours. The excess caustic liquor is removed by pressing until the press weight of the wet alkali cellulose is about 250 to 300 parts. The alkali cellulose is then shredded for 2 to 3 hours and then aged at 20° C. for 50 to 60 hours. The aged alkali cellulose is then xanthated, according to any well-known means, at 25° C. for 2 to 3 hours, using 33 parts of carbon bisulphide. The viscose solution is made by dissolving the cellulose xanthate in caustic soda solution at 18° C. to produce a solution containing 7% cellulose and 6% alkali. This viscose is ripened at 18° C. to a salt index of 8.0 (see Reinthaler and Rowe, "Artificial Silk", 1928, page 68), at which index it is spun.

The viscose solution is conducted to the spinning room through nickel pipe lines, care being taken to prevent contamination in any form, and is spun into a bath containing about 65% sulphuric acid which is maintained at a temperature of 10° C. A small bobbin-type spinning machine is used, such as is manufactured by the Oscar Kohorn Co.

In the operation of the present process, and as diagrammatically illustrated in Figure 1, the spinning trough is divided into two compartments 1 and 2 by a partition 3 running lengthwise on the machine. The front compartment 1 is filled with the acid-coagulating bath. The rear compartment 2 is filled with water maintained at a suitable temperature for immediately quenching the strong acids.

A thread 4 is conducted a distance of 20 inches from a nozzle 5 to a floating roller 6 of the type described in Patent 1,878,455, and is led around this roller, and then returned about 5 inches to another floating roller 7 which serves to apply more tension and also to change the direction of travel of the thread. From the roller 7 the thread is led to a rubber-covered rod 8, up and across the partition 3, separating the acid from the water bath. The thread travel in the water bath totals about 11 inches with the water flow preferably countercurrent to the direction of the travel of the thread. Further tension is applied to the thread during its travel through the water bath by the use of two or more floating roller guides 9 and 10, as referred to above. After leaving the roller 10, the thread 4 passes under a rubber-covered hook guide 11, thence over a

traversing guide and onto a bobbin (not shown).

The thread is wound on a bobbin at a speed of 3,000 inches per minute and under a final tension of about 0.4 gram per denier. The thread spun in this example is 100 denier-40 filament, so that the total tension applied is about 40 grams. Following the spinning operation, the thread is washed free from all traces of acid and dried in the customary manner. If desired the thread may be desulphured and bleached prior to drying.

Example 2.—Viscose, as is prepared in Example 1, is spun at a salt index of 8, using the bucket-type spinning machine instead of the bobbin-type, and spun into a spinning bath containing 64% to 72% acid. In this case, the thread 12, as diagrammatically illustrated in Figure 2, is led from a spinning nozzle 13, a distance of about 20 inches, to a rotating guide 14 of the type described in British Patent No. 318,258. The thread passes around the rotor guide and then passes on to the feed wheel 15, then around a small roller guide 16, under the feed wheel and again over the feed wheel from which it passes to a bucket not shown. This feed wheel construction is shown in patent application Serial No. 411,786. The guide 16 runs in a water bath 17 in order to quench the strong acid and then, after again passing around the feed wheel, the thread is conducted into the spinning bucket in the customary manner. The tension on the thread as it passes from the roller guides to the feed wheel is 0.14 to 0.25 gram per denier or, in the case of 100 denier-40 filament thread, 15 grams to 25 grams.

Example 3.—The mode of operation in this case is the same as in Example 1, except that 15 parts of carbon bisulphide per 100 parts of cellulose are used in preparing the viscose. Further, the viscose is not ripened to a salt index of 8, but is spun immediately after mixing, or within a 10 to 12 hour period of ripening at 18° C. following the mixing operation. The bath, in this case, contains 47% instead of 65% sulphuric acid. Other conditions are the same as in Example 1.

By the process which I have described, it is not difficult to produce artificial threads of uniform and good quality having a dry tenacity of more than 2 grams per denier, or even more than 3 or 4 grams per denier, and more than 2 grams per denier in the wet state.

The threads so produced are not only substantially free from broken filaments and fluff, but are much more uniform in tenacity, elongation and dyeing characteristics than high tenacity threads produced heretofore in plasticizing baths.

Although the viscose which I have specified in my examples is prepared from wood pulp, it may, of course, be made from cotton, cotton linters, hull fiber, or any other suitable cellulosic material. Further, it need not be handled with the care and equipment set forth in my copending application Serial No. 676,463, but may, of course, be handled in the customary manner although the results will be somewhat poorer.

It is obvious that the process may be conducted in ways other than mentioned above, since the variables involved are interdependents and several of these variables may be concurrently varied without affecting the nature of the product. For instance, viscose containing larger amounts of caustic may be spun in baths of higher acid concentration. Unaged or only slightly aged alkali cellulose yields viscoses which require stronger acid for their plasticization. The strength of the acid is also dependent upon the amount of cellulose in the viscose and upon the salt index to which the viscose is ripened. Such factors as bath travel, tension applied to the thread at different stages in its travel, method of washing, or the agent used for quenching may be changed without departing from the spirit of my invention.

Though the invention has been particularly described in connection with the manufacture of artificial thread, it is not restricted thereto. It is equally applicable to the manufacture of ribbons, films, straw, horsehair, etc.

I claim:

1. A method of producing artificial filaments, threads, straw, horsehair, ribbons, films, etc., which comprises extruding a viscose solution into a bath containing at least 45% sulphuric acid, imposing a substantially uniform and constant tension of at least 0.1 gram per denier and without any undue sliding friction on the structure intermediate the extruding device and draw-off device, checking the action of the acid on the structure prior to the production of any deleterious results, and winding the structure in an operation combined with the production of the same at a speed not less than 3,000 inches per minute.

2. A method of producing artificial filaments, threads, straw, horsehair, ribbons, films, etc., which comprises preparing a viscose solution free from deleterious contaminations, delivering said viscose solution without changing the quality thereof to the extruding device, extruding the viscose solution into a bath containing at least 45% sulphuric acid, imposing a substantially uniform and constant tension of at least 0.1 gram per denier and without any undue sliding friction on the structure intermediate the extruding device and draw-off device, checking the action of the acid on the structure prior to the production of any deleterious results, and winding the structure in an operation combined with the production of the same at a speed not less than 3,000 inches per minute.

3. A method of producing artificial filaments, threads, straw, horsehair, ribbons, films, etc., which comprises preparing a viscose solution free from deleterious contaminations, delivering said viscose solution through equipment which is not corroded by said viscose and does not contaminate said viscose to the extruding device, extruding the viscose solution into a bath containing at least 45% sulphuric acid, imposing a substantially uniform and constant tension of at least 0.1 gram per denier without any undue sliding friction on the structure intermediate the extruding device and draw-off device, checking the action of the acid on the structure prior to the production of any deleterious results, and winding the structure in an operation combined with the production of the same at a speed not less than 3,000 inches per minute.

4. A process of preparing artificial filaments, threads, straw, horsehair, ribbons, films, etc., which comprises preparing a viscose solution having the desired salt index, delivering said viscose solution without changing the quality thereof to the extruding device, extruding the viscose solution into a bath containing at least 45% sulphuric acid, imposing a substantially uniform and constant tension of at least 0.1 gram per denier and without any undue sliding friction on the

structure intermediate the extruding device and draw-off device, checking the action of the acid on the structure prior to the production of any deleterious results, and winding the structure in an operation combined with the production of the same at a speed not less than 3,000 inches per minute.

5. A method of preparing artificial filaments, threads, straw, horsehair, ribbons, films, etc., which comprises preparing a viscose solution of the desired salt index, delivering said viscose solution through equipment which is not corroded by said viscose and does not contaminate said viscose to the extruding device, extruding the viscose solution into a bath containing at least 45% sulphuric acid, imposing a substantially uniform and constant tension of at least 0.1 gram per denier and without any undue sliding friction on the structure intermediate the extruding device and draw-off device, checking the action of the acid on the structure prior to the production of any deleterious results, and winding the structure in an operation combined with the production of the same at a speed not less than 3,000 inches per minute.

6. A method of preparing artificial filaments, threads, straw, horsehair, ribbons, films, etc., which comprises preparing a viscose solution having a salt index of 8, delivering said viscose solution without changing the quality thereof to the extruding device, extruding the viscose solution into a bath containing a high concentration of sulphuric acid, imposing a substantially uniform and constant tension of at least 0.1 gram per denier and without any undue sliding friction on the structure intermediate the extruding device and draw-off device, checking the action of the acid on the structure prior to the production of any deleterious results, and winding the structure in an operation combined with the production of the same at a speed not less than 3,000 inches per minute.

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