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ARTIFICIAL FIBERS

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Our present invention relates to the production of artificial filaments, fibers or ribbons from cellulose and more particularly to the production of curly filaments and the like.

In producing artificial filaments and the like, especially staple fibers, it is necessary to give the filaments a more or less fine-wavy stable curl, in order to render them wool-like. Not only is this curl of the artificial filaments of importance for the properties and working up of products unspun, but for the properties and particularly the strength of the yarns spun therefrom.

Many properties have been made for curling staple fibers and the like. However, there is no method which results in products satisfactory in every respect. It has, for instance, been proposed to curl the cut fiber by means of liquids intensely moved, for instance, after-treating baths or decomposing baths. It is also possible to press filaments freshly produced and still plastic as well as finished, for instance, with fluted rolls to form fine waves. Moreover, it has been proposed to give loose fibers a wavy form by a treatment with vapors or by alternately cooling and heating them. Furthermore continuous filaments may be waved by an apparatus in which the filament freshly spun is conveyed towards a fixed scraping device. It is also known to curl the fibers by means of shaking, motion of the spinning nozzle or vibrations of the spinning bath. A process has also been proposed in which filaments of cellulose xanthogenate are converted into cellulosehydrate in hot decomposing liquids without stretching. It is finally known to subject the filaments in a strongly acid condition after being treated in the spinning bath to a stretching procedure under a stress gradually increasing in several drawing frames, then to deacidify the filaments with stretching at a temperature between 90 and 100° C, and to cut them into staple fibers in a desulfurizing liquid whereupon they are after-treated in loose form.

All these modifications are of disadvantage as they cause much trouble, in addition the curl obtained is not sufficiently stable and the quality of the resulting filaments and the yarns made therefrom is decreased by these curling methods.

Our present invention has as an object to overcome the disadvantages mentioned above.

Another object is the provision of a process for producing a permanent curl without carrying out troublesome steps.

A further object is to provide textile fibers valuable respecting their appearance and working up as well as the properties of the yarns spun therefrom.

Yet another object is to provide cut bundles of curled fibers retaining their parallel position.

Other objects of our invention will become apparent from reading the detailed description following hereinafter.

These objects are accomplished by preparing filaments, fibers or ribbons which entirely consist of cellulosehydrate and are formed by two superimposed layers exhibiting structures different from each other. This difference in structure is produced by regenerating to cellulosehydrate one of these layers before stretching and the other layer during or after stretching. The layers react in a different manner when treated with swelling agents preferably heated and would extend to different lengths if possible. Since the two layers, however, strongly adhere to each other, they are forced to warp more or less. Thus filaments are obtained which are curly and wavy. As a rule the outer layer is regenerated without or with slightly stretching, whereas the regeneration of the inner layer is effected with strongly stretching. However, it is also possible to attain curl in the filament, if the outer layer is treated under strong drawing and the inner layer without drawing.

The formation of an outer layer not stretched or only slightly stretched is effected in working up viscose according to this invention in weakly acid baths, the acid content of which may be dependent upon the ripeness of the spinning solution and the desired titer of the filament to be spun, but must not be substantially higher than that of sulfuric acid of 12% strength.

The regeneration and intense stretching of the inner layer of the filaments is carried out in a hot water bath which may contain small amounts of acids or acids and salts. It is of advantage to use two water baths, the first containing some acid and, if desired, salt and the second only water.

In order to obtain the difference in tension the continuous or cut two-layered filaments are swelled without stretching in preferably hot water, whereby the filaments freshly produced are simultaneously purified. In addition, the employment of hot water is favorable, because the tensions are more rapidly released in the heat than in the cold. Instead of water there are also used other liquids capable of swelling cellulose, for instance, cold and hot solutions of salts, alkalies or acids, especially solutions of substances capable of chemically acting on the swelling cellulose, such as zinc chloride, calcium thiocyanate, sodium hydroxide, sodium sulfide or strong sulfuric acid. Steam is also suitable for producing curl.

It is advantageous to swim the filaments in a loose condition in the swelling liquids, in order that each filament can move as freely as possible. An intense moving of the liquids is to be avoided since nubs can be formed upon the filaments thereby. It is desirable to treat the filaments freshly produced and being still wet in the swelling baths. However, the treatment may also be carried out by causing threads already after-treated with drawing and dried to swell. In this case the success generally is not great.

Processes, in which filaments spun in acid baths without stretching have been regenerated while strongly stretching, do not yield curl in the filaments on after-treating with swelling agents. The reason resides in the fact that the filaments when treated with the swelling agents have not been capable to be curled or have not possessed the necessary difference in tension between the outer and the inner layers or the outer layer has been too thick or too thin compared with the inner layer. As we have found it is necessary that the tendencies to shrink of the two layers are different from each other but approximately equal in strength. It is without difficulty to control the procedures of the coagulation and regeneration so as to obtain the desired character of curl. The ripeness of the viscose can be chosen for a given effect of coagulation and a certain duration of action in the first bath so, that the filament is regenerated to such an extent after having passed through the first bath and the open air behind it that an approximately equal part, which is regenerated during or after stretching, remains for the inner layer. It is immaterial whether the non-stretched or only slightly stretched outer layer and the stretched inner layer are regenerated in one or several baths. If two baths serve to form the outer layer, the first coagulating bath may contain such substances as convert the surface of the filament only into cellulose xanthogenate. It is of advantage to stretch the inner layer as intensely as possible. The stretching is therefore conveniently carried out in a hot liquid.

When the ripeness and viscosity of the spinning solution are given in the spinning process according to this invention, the effects of coagulation and regeneration or the duration of action of the baths, i. e. the acid and salt content and the temperature of the baths, and the length of the paths in bath and air may also be controlled in such a manner that the energies produced in the two layers by releasing the difference in tension are different and act against each other, whereby a curl is obtained. The adjustment of the first bath is considerably facilitated by adding large amounts of salts capable of retarding the decomposition of the filament. There are cations suitable for producing a fine-wavy curl such as zinc, potassium, lithium, ammonium, aluminium, and magnesium. In order to find out a bath containing a favorable combination of acids and salts, it is merely necessary to place the spun filaments into hot water and observe the forming curl. By varying the acidity of the first bath and/or the salt content and/or the period of action of the baths the conditions are quickly determined on which the most suitable curl is produced. A short wavy curl is easily attained by using zinc and/or potassium salts.

The process of this invention is not only applicable to the production of cellulosehydrate filaments from viscose but to that of artificial filaments from copper hydroxide ammonia solutions. In this case the outer layer may be formed in the spinning funnel without considerably stretching and the inner layer subsequently outside the funnel during or after strongly stretching, preferably in hot water which may contain some acid. After the copper has been washed out from the thread, conveniently under stretching, curl is effected by the same means which are used for working up viscose.

For producing curl according to the present invention it is not necessary that the outer cellulosehydrate layer is sharply separated from the inner layer. Several intermediate layers may also be provided.

The more detailed practice of this invention is illustrated by the following examples. There are of course many forms of the invention other than these specific embodiments.

Example 1

Viscose containing 8% of cellulose and 6.5% of caustic soda with a ripening point of 7½ is extruded through a 2500/0.07 nozzle into a bath containing 7.9% of H_2SO_4, and 29.5% of Na_2SO_4 and kept at 50° C, the length of immersion being 15 cm., to form a 1.4 denier thread. The bundle of filaments is collected over a guide roller on a drum having a peripheral speed of 33 m/minute and is continuously transferred through a water bath heated to 95° C and comprising a length of immersion of 1 m to a second drum having a peripheral speed of 55 m/minute. The threads are then sent through a cutting device without stretching. The staple fibers thus obtained are subsequently treated in a water bath at 80° C, in order to release the difference in tension, and remain therein floating free from one another until contraction is complete. Finally the staple fibers while conveyed by a band permeable to liquids are sprinkled with solutions suitable for after-treatment without being suspended in the liquids.

Example 2

Viscose containing 7.7% of cellulose and 6.5% of caustic soda with a salt point of 7¾ is extruded through a 600/0.08 nozzle into a bath containing 9.6% of H_2SO_4, 28.5% of Na_2SO_4 and 2.2% of $ZnSO_4$ and maintained at 50° C, the length of immersion being 15 cm., to form a 3.75 denier thread. This thread is transferred into a channel 1 m in length and containing water of 96° C by a drum at the rate of 29 m/minute and is stretched therein by a second drum having a peripheral speed of 55 m/minute. The fibers cut up without tension are then treated, without being previously dried, with sulfuric acid of 2% strength at 70° C to produce curl, are subsequently deacidified without being suspended in the liquid used, then desulfurized, soaped and finally dried. The fibers exhibit a good short-wavy curl, a tenacity (dry) of 3 grams per denier, a tenacity (wet) of 2 grams per denier, and an elongation of 19%.

Example 3

Viscose containing 7.7% of cellulose and 6.5% of caustic soda with a salt point of 7¼ is extruded through a 2500/0.07 nozzle into a bath containing 8.0% of H_2S_4, 27.3% of Na_2SO_4 and 1.8% of $ZnSO_4$ and kept at 50° C, to form a 1.4 denier thread, the length of the filaments immersed being 30 cm. The filament bundle is then collected on a drum having a peripheral speed of 34 m/minute and is continuously transferred through a water bath 4 m in length to a second drum heated at 80° C and having a peripheral speed of 55 m/minute. The filaments are then cut up without tension and treated with water in a vat at 75° C. The staple fibers thus curled and loosened are finally brought by a conveying band to the several treating baths.

Example 4

Viscose containing 7.7% of cellulose and 6.5% of caustic soda with a ripening point of 6¾ is extruded through a 2500/0.07 nozzle into a bath containing 8.7% of H_2SO_4, 21.6% of Na_2SO_4, 6.5% of $(NH_4)_2SO_4$, and 1.8% of $ZnSO_4$, the length of the filaments immersed being 15 cm. The bundle of filaments is collected on a drum at the rate of 32 m/minute and transferred through a water bath 3 m in length at 95° C while drawn by a second drum having a peripheral speed of 55 m/minute. The filament bundle is then cut up and the staple fibers are subsequently treated with sulfuric acid of 1% strength at 85° C, whereby the curl is released. Finally the fibers are freed from acid by washing, after-treated without being suspended in the used liquids and dried.

Example 5

Viscose containing 7.98% of cellulose and 6.36% of caustic soda with a ripening point of 7¼ is extruded through a 2500/0.07 nozzle into a bath containing 7.9% of H_2SO_4, 22.3% of Na_2SO_4, 6.7% of K_2SO_4, and 2.4% of $ZnSO_4$ and kept at 50° C, to form a 1.4 denier thread, the length of immersion being 15 cm. The thread is collected on a drum having a peripheral speed of 32 m/minute and is transferred through a hot bath 50 cm. in length and containing sulfuric acid of 2% strength and subsequently through a water bath 4 m in length at 90° C. The filaments are stretched by a second drum with a peripheral speed of 55 m/minute during these two procedures. The bundle is then cut up without drawing and treated with hot water to release the difference in tension. The staple fibers thus curled and loosened are finally after-treated and dried. The resulting fibers exhibit an especially fine and uniform curl and resemble merino-wool.

Example 6

Viscose containing 7.7% of cellulose and 6.5% of caustic soda with a salt point of 7½ is spun into a setting bath containing 7.9% of H_2SO_4, 26.8% of Na_2SO_4 and 2.4% of $ZnSO_4$ in a manner as described in Example 1. The filament bundle is collected on a drum having a peripheral speed of 33 m/minute and is continuously transferred through a hot bath 2 m in length and containing sulfuric acid of 2% strength and subsequently through a hot water bath 3 m in length, the filaments being drawn at the rate of 55 m/minute when treated in the baths. The fixed filament bundle is cut up and then treated in a bath containing a diluted solution of Na_2SO_3 to produce curl. The desulfurization effected thereby renders the usual desulfurizing stage unnecessary.

Example 7

Viscose containing 8.0% of cellulose and 6.5% of caustic soda with a salt point of 7.3 is extruded through a 800/0.08 nozzle into a bath containing 8.8% of H_2SO_4, 23.3% of Na_2SO_4, 6.2% of $LiSO_4$, and 2.0% of $ZnSO_4$ and kept at 50° C to form a 3¾ denier thread, the length of immersion being 15 cm. The filament bundle is collected on a drum having a peripheral speed of 29 m/minute and is stretched by a second drum having a peripheral speed of 55 m/minute. Between the two drums the bundle is transferred through a bath 15 cm. in length and containing sulfuric acid of 2% strength at 90° C. The filaments cut with or without tension are especially fine-wavy.

Example 8

Viscose containing 7.4% of cellulose and 6.5% of caustic soda with a ripening point of 7.0 is extruded through a 2500/0.07 nozzle into a bath containing 8.6% of H_2SO_4, 24.7% of Na_2SO_4 and 5.7% of $Al_2(SO_4)_3$ and kept at 50° C to form a 1.4 denier thread, the length of immersion being 15 cm. The bundle of filaments collected at the rate of 36 m/minute is transferred through a water bath 2 m in length at 95° C and stretched therein at the rate of 55 m/minute. The filaments now entirely consisting of cellulosehydrate are cut and soaked in hot water, whereby the curl is released. The fiber has a tenacity (dry) of 2.1 grams per denier and an elongation of 19% and exhibits a good curl.

Example 9

Viscose containing 7.4% of cellulose and 6.5% of caustic soda with a ripening point of 7.0 is extruded through a 2500/0.07 nozzle into a bath containing 8.5% of H_2SO_4, 24.4% of Na_2SO_4 and 6.2% of $MgSO_4$, to form a 1.4 denier thread, the length of immersion being 15 cm. The bundle of filaments collected at the rate of 35 m/minute is transferred through a water bath 2 m in length at 95° C and stretched therein at the rate of 55 m/minute. The bundle now entirely consisting of cellulosehydrate is cut up and treated with hot water to release the difference in tension.

Example 10

Viscose containing 7.7% of cellulose and 6.5% of caustic soda with a salt point of 3.75 is extruded through a 2500/0.07 nozzle into a bath containing 6.5% of H_2SO_4, 22.4% of Na_2SO_4, 6.4% of K_2SO_4 and 1.9% of $ZnSO_4$ and maintained at 20° C. The bundle of filaments collected at the rate of 45 m/minute is transferred through a bath 1m in length and containing sulfuric acid of 2% strength and then through a water bath 3 m in length at 95° C, the bundle being stretched at the rate of 55 m/minute when treated in the baths. The filaments are then cut up and treated with water of 80° C.

Example 11

Viscose containing 8.0% of cellulose and 6.6% of caustic soda with a ripening point of 7½ is extruded through a 2500/0.07 nozzle into a bath containing 9.2% of H_2SO_4, 21.5% of Na_2SO_4, 6.6% of K_2SO_4 and 2.1% of $ZnSO_4$, to form a 1.6 denier thread, the length of immersion being 15 cm. The bundle of filaments collected at the rate of 45 m/minute is transferred through a hot water bath 5 m in length and stretched therein at the rate of 55 m/minute. The releasing of the difference in tension of the filaments and the after-treating thereof may be carried out in a manner as described in any of the foregoing examples. By the increase in speed of 10 m the fiber is only slightly curled but obtains a high coefficient of elongation.

Example 12

Viscose containing 7.7% of cellulose and 6.5% of caustic soda with a salt point of 7¾ is extruded through a 600/0.08 nozzle into a bath containing 8.2% H_2SO_4, 27.8% of Na_2SO_4 and 2.1% of $ZnSO_4$ and kept at 50° C, to form a 3.75 denier thread, the length of immersion being 15 cm. The bundle of filaments is collected on a drum having a peripheral speed of 29 m/minute and stretched by a second drum having a peripheral speed of 55 m/minute. Between the drums the bundle is transferred through a bath 50 cm. in length and containing sulfuric acid of 2% strength at a temperature of 70° C and subsequently through a water bath 4.80 m in length and kept at 95° C. The bundle is then treated with water of 80° C without stretching to release the difference in tension and is subsequently brought to the several after-treating baths likewise without stretching, wherein the treatment is preferably carried out according to the dipping process.

Example 13

Viscose containing 7.7% of cellulose and 6.5% of caustic soda with a salt point of 7¾ is extruded through a 2500/0.07 nozzle into a bath containing 11.8% of H_2SO_4, 12.8% of Na_2SO_4, 6.3% of K_2SO_4, and 1.9% of $ZnSO_4$ to form a 1.8 denier thread, the length of immersion being 10 cm. The bundle of filaments is collected on a drum having a peripheral speed of 32 m/minute and stretched by a second drum having a peripheral speed of 55 m/minute. Between the drums the filaments are transferred through a bath 1 m long and containing hot sulfuric acid of 2% strength and subsequently through a water bath 5.0 m long and kept at 90° C. After the bundle has been cut up the curl is produced and the after-treatment carried out in a manner as described in any of the foregoing examples.

A similar curl is obtained if the setting bath contains only 10.9% of H_2SO_4 instead of 11.8% and is 15 cm. long instead of 10 cm. The fibers, however, do not exhibit curl, if 15% or 16% of H_2SO_4 are used instead of 11.8% in the bath.

Example 14

Viscose containing 7.4% of cellulose and 6.5% of caustic soda with a ripening point of 6½ is extruded through a yet with 60 holes, each of 0.07 mm diameter into a setting bath containing 8.3% of H_2SO_4, 20.3% of Na_2SO_4 and 4.2% of $ZnSO_4$ and maintained at 50° C to form a 2 denier thread, the length of the thread immersed being 15 cm. The filament bundle is collected on a drum having a peripheral speed of 30 m/minute and is continuously transferred through a bath 1.6 m in length and consisting of an aqueous solution of 1.5% of H_2SO_4, 3.0% of Na_2SO_4 and 0.4% of $ZnSO_4$ at 90° C, the stretching being effected by a drum having a peripheral speed of 56 m/minute. A good curl is produced by treating the continuous thread still wet with water of 90° C without stretching.

Example 15

A curl is not attained if in the modification described in Example 14 the setting bath contains 9.5% of H_2SO_4 instead of 8.3%. However, curl is again produced in this instance, if a yet with 1200 holes, each of 0.07 mm diameter is used.

The path in open air between the acid and the hot water baths is 4 m in all examples mentioned above; however, the paths may also be shorter or longer, for instance such as 20–50 cm or 6 m. If necessary, a too long path is compensated by reducing the concentration of acid in the first path or shortening the path in the bath.

Example 16

A copper hydroxide ammonia cellulose solution containing 9% of cellulose, 10% of ammonia and 5.4% of copper hydroxide is spun through a nozzle with 30 holes, each of 0.5 mm diameter into water in the spinning funnel at 30° C. The filament bundle leaving the lower end of the funnel is collected in a trough on a drum, the speed of which corresponds to that of the bundle extruded. The trough in which the lower part of the funnel immerses is filled with water from the funnel until the drum is below the surface of the liquid. The filament bundle is then transferred over a second drum non-driven into a further trough containing sulfuric acid of 2% strength and a temperature of 80° C. In this acid trough the bundle is stretched between two or several motor-driven drums, the length of immersion being 2 m. The extent of the stretching thus produced is 25%. The bundle is then cut up and the staple fibers are subsequently treated in a bath with water of 90° C without stretching and allowed to shrink.

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