Patented Aug. 22, 1944

2,356,518

UNITED STATES PATENT OFFICE

2,356,518

PROCESS FOR THE PRODUCTION OF WOOL-LIKE RAYON

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No Drawing. Application March 12, 1943, Serial No. 478,913. In Switzerland March 27, 1942

7 Claims. (Cl. 57—157)

This invention relates to a process for the production of wool-like rayon.

In accordance with a known process, wool-like rayon is produced by taking twisted or not twisted rayon yarns of regenerated cellulose and winding them on bobbins with a twist which is considerably higher than the normal twist; then they are moistened in highly twisted condition at high temperature, and thereupon untwisted beyond the zero point. By subsequently leaving the yarns in untensioned condition or by subsequently steaming in untensioned condition, the yarn is given a durable, strongly curled (crimped) wool effect (see U. S. Patents Nos. 2,019,183 and 2,019,185).

It has now been found that the quality and the durability of the products obtained can be still further increased, and further valuable properties can be imparted to them if one submits the relatively highly twisted rayon yarns of regenerated cellulose to a treatment with strong swelling agents, in other words treated with a chemical medium capable of rendering the yarn plastic and for a length of treatment until the yarn is plastic whereupon they are twisted back (untwisted) beyond the zero point, after washing with or without intermediate drying which is preferably carried out at increased temperature. In the practice of the present invention utilizing strong swelling agents, the subjecting of the yarn to high temperature while in highly twisted condition (as was necessary with the prior processes) may in many instances be dispensed with as will be seen from the examples hereafter given.

A further advantage of the manner of treatment in accordance with the invention consists therein that certain further treatments, as for example, dyeing or delustering (dulling) are simplified, in connection with which, at the same time, especially during the dyeing, a uniform dyeing, a deeper initial dyeing, and in this way, a saving in dyestuff are obtained.

One of the improvements in quality, due to the treatment in accordance with the invention is shown among other things by an increase in the elasticity of the curled (crimped) yarn. The improvement in the permanence of the crimping (curling) can be proven in a convincing manner by determining the so-called "permanent crimp."

The original length a of the crimped yarn is determined by measuring it in suspended condition. A load is then applied of 0.03 gram per denier and the yarn is submerged in a high glass beaker containing water at 60° C. for half a minute; then it is suspended and the length b of the loaded yarn is measured. Thereupon the yarn is dried in loose condition and one determines in the same manner as stated for a, the length c of the dried yarn. The "permanent crimp" in percent. is

$$\frac{b-c}{b-a}\times 100$$

Preferably, the average of about 10 tests is used.

It will be noted that the permanency of the crimp is very considerably increased by the treatment in accordance with the invention. This is especially shown by an improvement of the resistance of the crimp to laundering. A further advantage of the process is that due to the swelling agent treatment of the highly twisted yarns, the latter in loose condition show a greatly decreased tendency to become knotty, i. e., to form loops and to twist these loops. This is especially favorable when the highly twisted yarn is to be further used in the form of skeins or as single, continuous yarn.

As starting material one may use rayon yarns of regenerated cellulose, such as viscose or cuprammonium rayon yarns, in not twisted or pre-twisted condition, dulled or not dulled, and if desired also pre-dyed. While formerly the subsequent dyeing caused difficulties in connection with the manufacture of such wool-like rayon on account of the irregular initial dyeing, this need not be feared in connection with the new process, so that one need not hesitate to use undyed starting material which can subsequently be dyed as required.

The not twisted or pre-twisted rayon yarns are highly twisted—depending on the denier, to a greater or lesser extent—namely, to about 4 times the twist of the normal yarn or even more. This is done preferably on perforated spools (bobbins) made of material which is resistant to the swelling agents in question, if it is desired to effect the swelling of the highly twisted yarn on the spool. Hence spools may be used made, for instance, of metal or of plastic.

As swelling agents one may use the well-known customary strong swelling agents such as, for instance, caustic potash solution, caustic soda solution, zinc chloride, calcium thiocyanate. Sulphuric acid also may be used provided that the spools—if the treatment is effected on spools—are made of acid-resistant material. Depending on whether the swelling agent has only a swelling or also a parchmentizing effect, the "feel" and also the appearance of the yarn are influenced. In this connection it may be

noted that even when adding wetting agents or mercerizing auxiliary agents, if alkali solutions are used, the effect of the swelling agents on the highly twisted yarns takes place more slowly and less intensively than on normally twisted yarns, so that swelling agent concentrations can also be used which otherwise would be dangerous for rayon, for example, caustic soda solution of 10–18° Bé. Caustic potash solutions of 30° Bé. and also caustic soda solutions of 30° Bé. have proven to be especially effective swelling agents. However, other concentrations likewise, for instance from 6° Bé. upwards to above 30° Bé. can be used. Also solutions of zinc chloride or calcium thiocyanate of 60–70% are suitable and likewise lower concentrations if, for instance, higher treatment temperatures are selected. For caustic soda solution and caustic potash solution, a treatment temperature of about 80° C. has been found satisfactory. However, good results are also obtained at room temperature. The time of treatment is generally a few minutes, for example, 3 minutes with the caustic potash solution of 30° Bé. at 80° C.

The treatment with swelling agents can be advisedly carried out on perforated spools on which the high twisting was done, for instance by drawing or forcing the swelling agent through the perforated spools. One can, however, also unwind the highly twisted yarn and treat it with the swelling means in skeins under tension. The yarn can also be led continuously over guide rollers through a bath containing the swelling agent. After the treatment with swelling agent, the latter is advisedly washed out and the yarn dried, which can be done on the spools if the swelling agent treatment was also effected on same.

One may, however, also, depending on the kind of swelling agent, postpone the washing out until after the drying. The drying is effected advisedly at high temperature. After the washing and drying, the yarn is untwisted beyond the zero point, mostly to about 70 to 80 turns beyond the zero point in the opposite direction, whereupon the goods can be unwound, for instance, into skeins. The twisting back can be effected, if desired, without previous drying. If finishing (ennobling) treatments, such for instance as dyeing or dulling, are intended, they can be carried out advantageously after the treatment with swelling agent and between the washing and drying, and this can possibly be done on the spools. After twisting back beyond the zero point, the yarns can also be cut into staple fibers, and as such spun and further worked. In order to reduce their swelling properties one can also treat them after the untwisting with formaldehyde at high temperatures in the presence of acid catalysts, in accordance with any of the known processes. The following examples serve to explain the invention. They, however, do not by any means exhaust the different possibilities of carrying out the process.

Examples

Example I.—Viscose yarn of 450 denier is twisted in S-direction to 1500 turns per meter and wound directly on perforated tubes made of alkali-resistant material. The tubes with the twisted yarn wound on same are thereupon placed on a suitable creel and put in a dyeing apparatus as, for instance, the Obermayer or Franklin-Dyeing apparatus. Thereupon one causes caustic potash solution which has been preheated to 80° C. and the specific gravity of which at 15° C. is 30° Bé., to circulate through the tubes under pressure (as a rule ½ to 1 atmosphere gauge is sufficient for this purpose). After a time of action of 4 minutes, the caustic potash solution is drained from the tank and the yarn material is freed from the lye by washing with hot water. For a complete neutralization acidify with dilute acetic acid, wash well with cold water, then draw off the excess liquid by suction and finally dry the yarn by forcing hot air through the tubes. The yarn is then untwisted and twisted in Z-direction beyond the zero point to 70 to 90 turns per meter. The yarn in the form of skeins is then reeled and steamed without pressure, for 5 to 10 minutes. One obtains a strongly crimped voluminous yarn of good wool character.

Example II.—Rayon yarn which has been twisted, treated with lye and untwisted, as stated in Example I, is treated after reeling in skeins, for 15 minutes, at about 30 to 40° C. in a bath, containing 3 grams of oil soap per liter. It is then well rinsed, centrifuged and dried in freely suspended condition. One obtains in this way a strongly crimped wool-like yarn which is not essentially changed even by frequent washing.

Example III.—Rayon yarn which has been predyed with vat dyes of 300 denier is twisted in S-direction to 1800 turns per meter and inserted in the form of cross-wound bobbins on lye-resisting perforated cones or bobbins in an Obermayer dyeing apparatus. Thereupon at room temperature and for 10 minutes, caustic potash solution of 16° Bé. containing 10 cubic centimeters mercerizing Flerhenol (mercerization agent brought out by the firm of Fleschwerke Aktiengesellschaft, Frankfurt a. M., consisting of a mixture of crude cresol and fat solvents) per liter is caused to circulate through the apparatus, whereupon the yarn, after removal of the excess lye from the receptacle, is washed with water of about 60° to 70° C. in order to remove the lye. Thereupon acidify with dilute acid and wash until neutral. After the drying the yarn is twisted in the Z-direction beyond the zero point to 80 turns per meter. Two such yarns are thereupon twisted in the S-direction to 50 turns per meter, reeled and passed in the form of skeins through a bath containing 1 gram per liter of Igepon T (a sodium salt of the methyl taurides of higher fatty acids) at 50° C. for 10 minutes; then rinsed, centrifuged and dried in tension-free condition. One obtains in this manner a dyed, voluminous and soft yarn, the fibrils of which show a strong crimping or curliness. The effect has good resistance to laundering.

Example IV.—Viscose yarn of 400 denier is twisted in S-direction to 1600 turns per meter and directly wound on conical, perforated tubes. These cones, as described in Example I, are placed in a suitable creel, in a dyeing apparatus and treated in same for 3 minutes with a 23.5% caustic soda solution of 15° C. 10 cubic centimeters of mercerizing Flerhenol per liter or some other suitable mercerizing agent, as for instance Mercerol (a substance manufactured by Sandoz Chemical Works, Inc., of Switzerland, the principal ingredients of which are said to consist of "fractions of Xylenols"), is added in order to improve the wetting effect. After the draining off of the excess lye, thoroughly rinse with hot water and thereupon dye, using substantive dyestuffs, as for instance, diamine pure blue. Prepare a dyeing bath containing 1 gram Igepon

T per liter and dye at the boiling temperature, in connection with which the previously dissolved dyestuff is added slowly in different dosages to the dye bath. After obtaining the desired intensity of dyeing and a sufficiently satisfactory through dyeing, rinse well with cold water, draw off by suction and dry the yarn with hot air. The yarn is thereupon untwisted and twisted in the Z-direction to 60 turns per meter, then reeled and the skeins slowly steamed. The resulting yarn is very soft and voluminous and the individual fibrils are strongly crimped. Light laundering as customary in the case of directly dyed textiles does not affect the crimping effect for all practical purposes.

Example V.—Viscose yarn of 450 denier is twisted in the S-direction to 1400 turns per meter and in this highly twisted condition is pulled in the form of individual yarns through a bath of caustic potash solution heated to 50° C. of a specific gravity at 15° C., 16° Bé., then squeezed, dried in a hot air current of 60–70° C. and reeled. Thereupon the yarn material is washed in skein form with hot water; thereupon acidified with dilute acid in order to neutralize the remaining alkali, then rinsed well and dried. The washing and neutralizing are effected advisedly under light tensioning of the skeins. The yarn is now twisted back in the Z-direction beyond the zero point to 100 turns per meter, and thereupon twisted together with a viscose crepe yarn of 100 denier to 70 turns per meter in S-direction. This new yarn is well suited for the production of novel fabrics as it can be used either in a correspondingly loosely arranged warp or both as warp and filling material. The fabrics thus obtained are creped in a soap bath, and if desired, afterwards subjected to bleaching and dyeing, and dried under slight tension. The fabrics obtained are very elastic, voluminous and of wool-like character.

Example VI.—Cuprammonium rayon of 360 denier is twisted in S-direction to 1700 turns per meter and thereupon passed in the form of individual yarns through a bath containing caustic potash solution of a specific gravity of 14° Bé. at 15° C. The caustic potash solution is heated to 70° C. and in order to increase the wetting ability 10 cubic centimeters of mercerizing Flerhenol are added per liter. The excess lye is squeezed off and the yarn is introduced in the lye-removing bath preferably over a reel in order to increase the time of action of the lye. The time of action of the lye should be at least one-half to one minute. The lye is removed with hot water, then acidified for a short time with dilute acetic acid, and rinsed with cold water. Thereupon the yarn is squeezed, dried in a hot air current or by passing over hot cylinders and wound. The yarn is then twisted in Z-direction to 70 turns per meter, reeled and subsequently, in the form of skeins and in tensionless condition, soaped for 10 minutes at 50° C., rinsed, centrifuged and dried in freely suspended condition. One obtains in this way a voluminous wool-like yarn. The strong crimping of the individual fibrils brings about a diffused reflection of the light and the changed textile material shows only a light soft luster.

Example VII.—The cuprammonium yarn which has been highly twisted in accordance with the method described in Example VI and treated with caustic potash solution of 16° Bé. is washed and dried and then untwisted in the Z-direction to 40 turns per meter; then reeled, steamed for 10 minutes without pressure in tension-free condition and thereupon twisted together in the Z-direction with a normal twisted rayon yarn of 120 denier to 60 turns per meter. One obtains in this way an interesting mixed yarn which is very suitable for weaving. Instead of twisting the cuprammonium treated yarn with a normal rayon yarn one can also weave it in simultaneously side by side with an ordinary yarn.

Example VIII.—Viscose rayon of 450 denier is twisted as described in Example I, treated on tubes with hot caustic potash solution, then untwisted, reeled, and subsequently steamed. Thereupon the yarn is passed in the form of skeins for 10 minutes through a cold bath of the following composition:

	Cc.
Formaldehyde technically pure concentration	100
Aluminum thiocyanate solution 17° Bé	40
Water	860
	1000

The excess liquid is removed by centrifuging. The yarn is dried at 60 to 70° C. in loosely suspended condition and then heated for 15 minutes to 120° C. The resulting product excels by its exceptionally good resistance to laundering. It is very similar to wool with regard to its appearance as well as its reduced conductivity of heat and also to its absorption of water. On account of the reduced swelling property, the water-absorbing property is considerably reduced as compared with the starting material.

Example IX.—Viscose yarn of 450 denier is twisted to 1500 turns per meter in the S-direction, then wound directly on perforated tubes which are placed in the dyeing apparatus. Thereupon there is passed through the tubes under pressure at a temperature between 90 and 95° C. a zinc chloride solution obtained by the dissolving of 4 parts by weight of powdered zinc chloride in 6 parts by weight of water. After letting it act for 5 minutes, the zinc chloride solution is drained from the receptacle. The treated material is thereupon completely freed of the zinc chloride solution by washing with strongly diluted acetic acid. It is then well washed with cold water and dried on the tubes. Thereupon the yarn is untwisted and twisted in Z-direction beyond the zero point to 80 turns per meter. After reeling into skeins of about 30 grams, the material is steamed in tensionless condition for 5 minutes without any pressure. The yarn thus obtained has a good wool character, it is voluminous, soft and very elastic.

The term "relatively highly twisted yarn" or "relatively highly twisting yarn" as herein used is meant to define a twist which for any particular yarn is very substantially greater than its normal or customary twist. Except where otherwise so stated in any of the respective claims, it is to be understood that the claim is not to be limited to the drying step taking place prior to the detwisting.

While the invention has been described in detail according to certain preferred methods of carrying out the process, it will be obvious to those skilled in the art after understanding the invention, that changes and modifications may be made therein without departing from the spirit or scope of the invention, and it is intended in the appended claims to cover all such changes and modifications.

What is claimed as new and desired to be secured by Letters Patent is:

1. Process of producing wool-like artificial silk yarns of regenerated cellulose, which comprises taking such a yarn which has been relatively highly twisted and subjecting it to treatment with a strong swelling agent consisting of a chemical medium capable of swelling the yarn and continuing that swelling treatment until the yarn is appreciably swollen, thereafter washing the yarn, detwisting it beyond the zero point and drying.

2. Process according to claim 1 wherein the relatively highly twisted yarn after being subjected to the swelling agent washed and detwisted, is reeled off in the form of skeins and steamed.

3. Process according to claim 1 wherein the relatively highly twisted yarn is dyed after the treatment with the swelling agent and between the washing and drying.

4. Process for producing wool-like artificial silk yarns of regenerated cellulose, which comprises relatively highly twisting such a yarn on bobbins made of material resistant against a swelling agent to which the yarn is to be subjected, subjecting the yarn on such bobbins to treatment with a strong swelling agent consisting of a chemical medium capable of swelling the yarn and continuing that swelling treatment until the yarn is appreciably swollen, thereafter washing the yarn, detwisting it beyond the zero point and drying.

5. Process of producing wool-like artificial silk yarns of regenerated cellulose, which comprises taking such a yarn which has been relatively highly twisted and subjecting it to treatment with a swelling agent consisting of a chemical medium capable of swelling the yarn and continuing that swelling treatment until the yarn is appreciably swollen, thereafter washing the yarn, drying the yarn and then detwisting it beyond the zero point.

6. Process according to claim 1, wherein the relatively highly twisted yarn after being subjected to the swelling agent and washed and detwisted, is subjected while in a loose untensioned state, to moist heat.

7. Process according to claim 1, wherein the relatively highly twisted yarn is subjected to a dulling treatment after the treatment with the swelling agent and between the washing and drying.

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