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NEW METHOD OF MANUFACTURING ACRYLIC
FIBERS AND THE RELATED PRODUCTS

Kozo Yamada, Yasuo Saji, Ichiro Yamamoto, and Shigeru Ikegami, Shizuoka Prefecture, Japan, assignors to Toho Beslon Kabushiki Kaisha, Tokyo, Japan, a corporation of Japan

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1 Claim

ABSTRACT OF THE DISCLOSURE

A method for producing an acrylic fiber in which a spinning solution containing at least 85% (wt.) of polyacrylonitrile and zinc chloride is spun into an aquagel tow which is then washed free of the zinc chloride. The washed tow is then contacted with an aqueous emulsion containing an oiling agent after which the tow is dried, heat-stretched and then either heat-relaxed or heat-set.

The present invention relates to an improved process of spinning and after-treatment for the production of acrylonitrile polymer fibers and more particularly to the preparation of acrylic regular and shrinkable fibers of improved compositions. It has particular reference to the preparation of such acrylic fibers that their softness and anti-electrostatic property are excellent and maintained semi-permanently and that their knitted and woven goods give an excellent "hand" and have a high resilience comparable to that of wool goods. Another aspect of the invention is to facilitate the heat-stretch and heat-relaxation in the process of spinning and after-treatment for the production of acrylic fibers by using the spinning dope composed on an acrylonitrile polymer containing at least 85 weight percent of polymerized acrylonitrile and an aqueous salt solvent.

The method of the present invention consists of the following procedures in the process of producing acrylic fibers from the spinning solution composed of an acrylic polymer containing at least 85 weight percent of polymerized acrylonitrile and an aqueous salt solvent such as aqueous concentrated solution mainly of zinc chloride, sodium thiocyanate or calcium thiocyanate.

(a) spinning said acrylic polymer into an aquagel tow of filament and washing the aqueogel tow (to be construed herein as inclusive "filament") until substantially free of said solvent salt;

(b) contacting the washed aquagel tow of which fiber structure is substantially in an un-oriented condition with an aqueous emulsion containing an oiling agent or oiling agents selected from the surfactants of anionic, cationic, non-ionic and amphoteric types, so as to subject said aquagel tow to absorbing at least 1.5 weight percent in total of said oiling agent (to be construed herein as inclusive mixture of a plurality of agents) based on the resultant fiber in dry state; and

(c) subsequently drying said aquagel tow containing said oiling agent, heat-stretching the partially or substantially dried tow and heat-relaxing or heat-setting the stretched tow.

As well known oiling treatment in the process of acrylic fiber production has been usually done for the heat-stretched or heat-stretched and heat-relaxed fibers of which structure has been in substantially or at least partially oriented condition. The most part of the oiling agent adhered to the fiber is present on the fiber surface and can be removed easily with a hot alcohol-benzene mixture. The amount of the oiling agent to be added has

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been usually controlled to be in the range of 0.2–0.6 weight percent based on the dried fiber. Since the object of such an oiling treatment has been mainly to improve the physical property of fiber surface such as anti-electrostatic property and friction coefficient, there has been no practical demand for permeating a considerable amount of an oiling agent over 1 weight percent into fiber structure. In fact, with respect to the use of oiling agent in synthetic fiber industry, and considerable effect other than that of improving the practical property of fiber surface has been scarcely expected.

Contrary to the ordinary method of oiling treatment mentioned above the present invention is characterized by the facts that an oiling treatment is carried out for the aquagel tow which has not yet been heat-stretched and is in the condition of substantially un-oriented fiber structure and that the content of the oiling agent absorbed by the aquagel tow is controlled to be at least 1.5 weight percent based on the product fiber in its dry state. In this case, the oiling treatment is usually done by subjecting said aquagel tow to intimate contact with an aqueous emulsion of a synthetic surfactant, and the tow can absorb a considerable amount of the surfactant, for a very short time below one minute, due to its un-oriented aquagel fiber structure. The absorbed surfactant will be permeated into the aquagel fiber structure to form an acrylic fiber of a new composition.

The amount of the surfactant to be absorbed can be easily controlled by selecting or adjusting the surfactant concentration of the aqueous emulsion, the emulsion temperature and the contacting time of the aquagel tow with the emulsion, although the affinity or absorption tendency of the surfactant to said aquagel acrylic fibers is often dependent upon the chemical composition and molecular weight of the surfactant.

The present invention is based on the un-expected findings that such a special oiling treatment as mentioned above facilitates the subsequent heat-stretching and heat-relaxation and that the knitted and woven goods made from the acrylic fibers thus obtained given an excellent "hand" have a high resilience comparable to that of wool goods and keep their good handling property for a long time.

According to the present method, the acrylic tow subjected to absorption of a considerable amount of surfactant easily heat-stretched at a lower temperature and or to a higher extent than that formed without said oiling treatment, even when the tow is substantially dried and then heat-stretched in dry air. Especially, a marked effect of such absorbed surfactant is exhibited in relaxing the heat-stretched tow in heated air or steam under atmospheric pressure, i.e. with increasing the absorption content of surfactant in the fiber, the maximum shrinkage in relaxation at a given temperature and for a given period is considerably increased or the relaxation of a given shrinkage can be carried out at a lower temperature or in a shorter period.

Several examples of those effects are shown in Table 2 and Table 3.

Also, it is surprised to have found that the acrylic fibers produced through the spinning and after-treatment process according to the present invention are more bulky in "hand" than those produced by prior arts and that the knitted and woven goods made from the acrylic fibers produced by the present method give an excellent "hand" and have a high resilience comparable to or equal to that of wool goods and show only a negligible deterioration in their quality related to the "hand" even after several cycles of practical washing and drying, which are superior to those made from other acrylic fibers. In the present method, almost of the surfactant absorbed into

the aquagel fiber structure is irreversibly fixed by drying the resultant aquagel fiber in hot air or by heat-stretching the aquagel fiber at temperatures over about 80° C., which can not be extracted with hot alcohol-benzene mixture. Such a fixed surfactant is called as "inner oil" hereafter in this specification. The above mentioned effects of the present invention will be considered to be attributed directly or indirectly to the presence of such "inner oil." On the other hand, in the case of ordinary methods of acrylic fiber production, an aquagel tow is heat-stretched (and is necessary heat-relaxed) to have a substantially or at least partially oriented fiber structure and then subjected to an oiling treatment so as to have 0.2–0.6 weight percent of a surfactant (based on the dried product fiber), so that most of the surfactant adhered will be present on the fiber surface. Such an oiling agent can be washed out easily with a hot alcohol-benzene mixture, which is called as "surface oil" for short hereafter. Table 1 shows some typical results on the "inner oil" and "surface oil" for the acrylic fibers produced by the present invention and by the prior art, where the amount of the "inner oil" was estimated as the difference between those of the "total oil" and the "surface oil." The amount of the "total oil" was estimated from the measurement of the true consumption of the surfactant per unit weight of the product fiber, with correcting the material loss accompanied with the experiment, and the amount of the "surface oil" measured by an extraction method with a hot alcohol-benzene (1:2) mixture.

As shown in Table 1, the amounts of "surface oil" according to the present method is in the same range of or not considerably different from those for the fibers produced through an ordinary procedure of oiling treatment, which will be suitable for making spun yarns and filament yarns.

The "inner oil" has been found to cause no trouble in the processes of yarn spinning knitting and weaving.

TABLE 1

Oiling treatment	Exp. No.	"Total oil" wt. percent	"Surface oil" wt. percent	"Inner oil" wt. percent
Immediately after washing	1	1.85	0.21	1.64
the aquagel fiber with-	2	2.56	0.32	2.24
drawn from a spinning bath	3	4.50	0.40	4.10
(the present invention).				
After the heat-relaxation sub-	4	0.18	0.16	0.02
sequent to a steam-stretch-	5	0.25	0.22	0.03
ing the aquagel fiber (the	6	0.50	0.49	0.01
prior art).				

The product fibers obtained through the present method can be, therefore, subjected directly to the subsequent processes such as tow cutting, Turbo processing and yarn spinning. It may be often found that the amount of the "surface oil" resulting from the application of the present method will be not so enough that the product fiber can be directly supplied to yarn spinning machines to form spun yarns of good quality. In such cases, the two may be contacted again, after the heat-stretching and heat-relaxation, with an aqueous dilute emulsion of an oiling agent so as to increase the resultant amount of the "surface oil" up to the range suitable for yarn spinning.

As described before, the heat-stretch to be applied after drying an aquagel acrylic fiber and the subsequent heat-relaxation are facilitated when the fiber to be heat-stretched has a considerable amount of "inner oil." Some examples of the effect are shown in Table 2 and Table 3.

Table 2 shows an experimental result on the relationship between the "total oil" content and the maximum heat-stretching ratio, where the acrylic tow samples were prepared by a wet-spinning method using an aqueous zinc chloride solvent and dried in hot-air before the heat-stretching experiment. Table 3 represents the maximum shrinkage measured for each of the heat-stretched tows, corresponding to each of the experimental numbers in Table 2, where the heat-relaxation was carried out in a

steam which was under atmospheric pressure and over-heated to a high temperature near 240° C. It is clear from these results that both

TABLE 2

Sample	"Total oil" (wt. percent)	Maximum heat- stretching ratio, percent
A-1-----	0	350
B-1-----	1.5	450
B-2-----	3.0	580
B-3-----	5.1	600

TABLE 3

Sample:	Maximum shrinkage, percent
A-1 -----	10
B-1 -----	24
B-2 -----	30
B-3 -----	40

the maximum ratio of heat-stretching and the maximum shrinkage of heat-relaxation subsequent to the stretching increase considerably with increasing the amount of "total oil" or strictly speaking the "inner oil."

Practically, the heat-stretching over 4 times at a temperature between 100–180° C. and the subsequent heat-relaxation at a temperature over 150° C. will be preferable for producing the acrylic fibers of high bulkiness as well as good mechanical property.

The characteristic in quality of the knitted and woven goods made from the acrylic fibers produced by the present invention has been described already in this specification. However, such quality expressions as "excellent hand" and "high resilience comparable to that of wool goods" are not quantitative but rather subjective and sensory. It is therefore desirable to provide a testing method by which the effect of the present method on quality of the end-used products may be numerically estimated. Since there has been no instrumental method

of testing by which the "hand" and "resilience" of knitted and woven goods can be quantitatively detected, the present inventors applied a sensory inspection by a group of specialists having much experience on the quality test of fiber goods.

Its procedure and a typical inspection result on the effect of the present method will be described in Example 2 in this specification.

The present inventors have also found that a desired shrinkable fiber can be effectively manufactured by subjecting the acrylic regular tow made through the method as referred to above to some subsequent treatments.

As well known, acrylic shrinkable fibers can be manufactured by a process in which an acrylic regular tow is heat-stretched again in its dry state (hereafter this heat-stretch is called as "the second stretch" to discriminate it from the heat-stretch applied in the process of producing the regular tow), and then immediately cooled under tension in air stream, and if necessary subjected to cutting. Their shrinkage, which is measured as the degree of their shrinking in fiber length in boiling water or in steam at 100° C., tends to increase with increasing the second stretching ratio, but increase in the ratio results in decrease of the knot strength and knot elongation of the resultant fibers, which is not a desirable tendency from the viewpoint of manufacturing

good shrinkable yarns. Since industrial spinning operation with a flat or roller carding machine will demand a lower limit of their knot strength or knot elongation in order to produce good shrinkable yarns, there will be an upper limit of the second stretching ratio to be applicable and consequently their practical shrinkage will be limited.

In the case of the acrylic regular tows produced by a wet-spinning method using an aqueous salt solvent such as an aqueous concentrated solution of zinc chloride, thiocyanate or nitric acid, the fiber knot strength and knot elongation tend to more decrease by the second stretching than those of the acrylic tows produced using an organic solvent such as dimethyl formamide, so that there has been a demand for any improved method by which the shrinkable fiber of a given shrinkage can be produced at a smaller ratio of the second stretch. The present method has been found to meet such a demand. The shrinkage in percent of the shrinkable fiber manufactured from the regular tow produced by the present method is higher by 2–10% than those of the usual shrinkable fibers when all the conditions of the second stretching are fixed constant. For example, when the second stretch of 1.31 times was applied, the shrinkage was 14–15% for the shrinkable staple made from the acrylic tow with the present invention but in the range of 9–12% for those made from the acrylic tow produced in accordance with the prior art. For the second stretch ranging 1.4–1.5 times, the shrinkable staples of 21–24% shrinkage and 1.5–1.6 grams per denier of knot strength could be obtained from the acrylic tow with the present invention while for the shrinkable staples from the above ordinary tows the shrinkage was 14–16%.

In making acrylic shrinkable fibers the present inventors have found that an application of a weak relaxation with steam is very much effective to improve the uniformity of shrinkage of the product fibers although their shrinkage is lowered a little depending on the degree of the applied relaxation.

This result will be very important, because the variance in yarn shrinkage of shrinkable yarns depends largely upon the variance in shrinkage of the material fiber. The copulation standard deviation σ of shrinkage for a lot of acrylic shrinkable fibers is markedly decreased by applying a weak steam relaxation in the range of 2–6% to the tow subjected to the second stretch. For example, with respect to the acrylic shrinkable staple manufactured under the second stretch of 1.4 times, the mean shrinkage percent and σ were respectively 25% and 3.1% without undergoing the above-mentioned relaxation but were respectively 21% and very 0.9% after applied the steam relaxation of 4%; also, in the case of the second stretch of 1.5 times, the mean shrinkage and σ were respectively 30% and 3.3% without the treatment but respectively 24% and only 1.4% after 6% relaxation. In those experiments the material tows produced by the present invention contained 3.5 weight percent of "inner oil."

The surfactants usable for the present invention are of cationic, anionic, non-ionic or amphoteric type. Among of "cationic" surfactants, there may be alkyl amine acetate, acetate, alkyl pyridium chloride, alkyl trimethyl ammonium chloride, N-alkyl-N-ethyl morpholinium ethosulfate, diethanolamine alkyl ester and its neutralization compounds, bis N,N'-ethyl aminoethyl alkylamide, alkyl dimethyl benzyl ammonium chloride and polyoxyethylene alkyl amine, an alkyl radical of said cationic compounds preferably having 8 to 18 carbon atoms. Among of "anionic" surfactants, there may be sodium alkyl sulfate, polyoxyethylene alkyl sulfate, alkyl phosphate sodium dialkyl sulfosuccinate, sodium alkyl-N-methyl lauride and N-methyl alkyl glycine, an alkyl radical of said anionic compounds preferably having 5 to 20 carbon atoms. As non-ionic surfactants, may be used polyoxyethylene alkyl ether, polyoxyethylene alkyl

phenol ether, polyoxyethylene alkyl ester, polyoxyethylene alkyl amine, polyoxyethylene alkyl amide, polyoxyethylene alkyl amino amide, sorbitan mono-alkylate, glycerol mono-alkylate or condensation compounds of polyoxyethylene with polyoxypropylene, an alkyl radical of said non-ionic compounds preferably having 5 to 20 carbon atoms; and as amphoteric surfactants N-alkyl, N-dimethyl betaine or 1-hydroxyethylene-2-alkyl-2-imidazoline, an alkyl radical of said amphoteric compounds preferably having 5 to 20 carbon atoms.

A mixture of at least two surfactants belonging to any one of the four types (cationic, anionic, non-ionic and amphoteric types) is also employed.

Furthermore, a mixture constituted of at least two different types of surfactants is often used, except the combinations of cationic type with anionic type or amphoteric type and of anionic type with amphoteric type.

In the present invention, at least 1.5 weight percent of surfactant or surfactants based on the product fiber in dry state must be absorbed into an aquagel filamental fiber in a substantially unoriented condition.

The absorption content will be preferable in the range of 1.5–6 weight percent, although strictly speaking the chemical composition, molecular structure and molecular weight of the surfactant to be used will also depend on the above mentioned effects to some extent. The content in the range of 2.5–4.5 weight percent will be more preferable.

The present invention is effective when applied to the processes for acrylic fiber production in which an aqueous inorganic salt solution containing zinc chloride or an alkali or alkaline-earth metal salt of thiocyanic acid as a principal component is used as the solvent for fiber-forming acrylonitrile polymer or copolymer. It will be more effective for the aqueous saline solvent of zinc chloride alone or of a mixture of zinc chloride with a chloride of an alkali or alkaline-earth metal, where the total salt concentration is preferably in the range of 55 to 65 weight percent.

The acrylonitrile polymer employed in the present invention is polyacrylonitrile and any of the acrylonitrile copolymers containing at least 85 weight percent of polymerized acrylonitrile and up to 15 weight percent of other monomer or monomers copolymerizable with acrylonitrile such as methyl acrylate, methyl methacrylate, vinyl acetate, acrylamide styrene sulfonate, allyl sulfonate, methallyl sulfonate, acrylic or methacrylic acid, itaconic acid, vinyl pyridines and vinyl imidazoles.

EXAMPLE 1

A spinning solution comprising 90 parts of saline solvent containing 53 wt. percent zinc chloride, 4 wt. percent sodium chloride and 43 wt. percent water and 10 parts of acrylonitrile copolymer composed of 92 wt. percent acrylonitrile unit and 8 wt. percent methyl acrylate unit was spun out through 6,000 holes spinnerets into a coagulating bath of an aqueous salt solution prepared by diluting the saline solvent to 20 wt. percent concentration.

The aquagel tow withdrawn out of the coagulating bath was water washed until substantially free of zinc chloride where it was drafted by 2.5 times in the water baths to facilitate the washing. The washed aquagel tow was immersed in an aqueous emulsion at 40° C. for 15 seconds which contained 30 grams per liter of diethanolamine mono-ester of lauric acid phosphate, squeezed with press-rollers and dried. The dried tow contained 1.5 wt. percent of the surfactant. The dried tow was then heat-stretched by 5.2 times at 150° C., immediately relaxed by 30% in a steam over-heated to 200° C., cooled and crimped. The tow thus obtained had 0.2 wt. percent of the "surface oil" and 1.3 wt. percent of the "inner oil."

The knitted goods made from the fiber thus obtained gave an excellent "hand" and had a high resilience comparable to that of wool goods.

Their excellent "hand" was negligibly changed even after several cycles of their washing and drying. Such

were equal or comparable to those of bulky sweaters of 100% wool.

TABLE 3

"Hand" grade	Sample No.				
	1	2	3	4	5
	Amount of "total oil" in the fiber				
	0.36 wt. percent	1 wt. percent	2 wt. percent	3.5 wt. percent	4.5 wt. percent
Best.....	0 person.....	0 person.....	1 person.....	8 persons.....	1 person.
Very good.....	0 person.....	0 person.....	3 persons.....	2 persons.....	5 persons.
Good.....	0 person.....	1 person.....	5 persons.....	0 person.....	4 persons.
Medium.....	3 persons.....	6 persons.....	1 person.....	0 person.....	0 person.
Bad.....	7 persons.....	3 persons.....	0 person.....	0 person.....	0 person.

good results could not be obtained for those made from the acrylic fibers having no detectable amount of the "inner oil."

EXAMPLE 2

In the experiment A, the aquagel tow obtained under the same conditions as those in Example 1 was water washed, squeezed, immersed in an aqueous emulsion at 45° C. for 20 seconds, which contained 30 grams per liter of polyoxyethylene lauryl amine and 30 grams per liter of diethylaminoethyl stearyl amide, squeezed again and dried. The dried tow contained 3.5 wt. percent in total of the above surfactant mixture. The dried tow was heat-stretched by 5.0 times at 150° C., heat-relaxed in super heated steam at 200° C. and then contacted with an aqueous dilute emulsion of polyoxyethylene alkylamine so as to have additional 0.1 wt. percent of the alkylamine on the fiber surface. The total amount of the "surface oil" composed of polyoxyethylene alkylamine and diethylaminoethyl oleic acid amide was 0.35 wt. percent in total based on the knitted goods made from the product were very much wool-like in hand.

On the other hand, in the experiment B, the washed aquagel tow obtained under the same conditions as those described above was stretched by 5.0 times in hot water at 90-95° C., relaxed in boiling water, squeezed with press-rollers, contacted with an aqueous emulsion containing 7 grams per liter of polyoxyethylene alkylamine and 7 gram per liter of diethylaminoethyl oleic acid amide for 2 seconds and squeezed again. This wet tow was, then, dried under a very weak tension accompanying a further relaxation, subjected to heat-setting and a second oiling treatment with the alkylamine and finally dried. In this process the total relaxation applied to the tow was controlled to be of the same percent as that applied in the experiment A, and also the first and second oiling treatments were controlled so as to adjust the amount of the "surface oil" near 0.35 wt. percent in total. The product fiber had a good spinning property, but the knitted goods made from the fiber were inferior in their quality to those made in the experiment A.

Five tow samples containing different amounts of the oiling agent were produced under the same experimental conditions as described above, except that the total surfactant concentration in the first oiling bath was different for each sample making. From those tows, the corresponding five kinds of bulky sweaters were manufactured by applying the same processes and under all the same conditions, where the bulky yarns were made through Turbo process. For all the sweater samples, the knitting construction as well as the yarn count and shrinkage were all the same. A sensory inspection of the "hand" of those sweaters was carried out by 10 persons having a lot of experience on the quality test of knitted and woven goods. The statistical result shown in Table 3 was obtained, and an additional experiment showed that the "Best" and "Very good" grades indicated in Table 3

EXAMPLE 3

9 weight parts of copolymer composed of 92 wt. percent acrylonitrile and 8 wt. percent methyl methacrylate were dissolved in 91 weight parts of an aqueous saline solvent comprising 42 wt. percent zinc chloride, 16 wt. percent calcium chloride and 42 wt. percent water.

The polymer solution thus obtained was spun out through spinnerets into a coagulating bath of an aqueous salt solution prepared by diluting the saline solvent to 25 wt. percent concentration. The aquagel tow withdrawn out of the coagulating bath was water washed counter-currently, squeezed and immersed in an oiling bath of aqueous emulsion containing 20 grams per liter of dilauryl phosphate sodium salt, 20 grams per liter of polyoxyethylene stearic amide and 30 grams per liter of polyoxyethylene-polyoxypropylene block polymer.

The aquagel tow was then dried, heat-stretched by 5.5 folds at 150° C. and then immediately relaxed, by 35 percent based on the length of the stretched tow, in a steam overheated at 250° C. The resultant fiber contained 4.5 wt. percent in dry base of the surfactant. The knitted and woven goods made from the above fiber were more excellent in their handling quality and anti-electrostatic property than those made from acrylic fibers containing no detectable amount of any surfactant.

What we claim is:

1. A method for producing acrylic fibers from a spinning solution of an acrylic polymer comprising at least 85 percent by weight of polymerized acrylonitrile in an aqueous inorganic solvent comprising mainly zinc chloride, said method comprising:

- spinning said acrylic polymer solution into an aquagel tow and washing the resulting aquagel tow until same is substantially free from residual salt,
- treating the washed aquagel tow having a fiber structure which is substantially in an unoriented state with an aqueous emulsion of at least one surfactant selected from the group consisting of diethanolamine mono-ester of lauric acid phosphate, polyoxyethylene lauryl amine, diethylaminoethyl stearyl amide, dilauryl phosphate sodium salt, polyoxyethylene stearic amide and a block polymer of polyoxyethylene-polyoxypropylene, at a temperature below 50° C. so as to impregnate said washed aquagel tow with 1.5 to 6 weight percent of said surfactant based on the resultant dried fiber,
- subsequently drying the aquagel tow treated with said aqueous surfactant emulsion at a temperature above 80° C. to irreversibly fix said surfactant in the fibrous structure, and then
- heat-stretching the substantially dried tow over 4 times at a temperature between 100 and 180° C. and heat-relaxing the stretched tow with a superheated steam at a temperature of at least 150° C. under atmospheric pressure.

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5 JULIUS FROME, Primary Examiner

J. H. WOO, Assistant Examiner

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