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WOOL SUBSTITUTES AND PROCESS OF
SPINNING SHORT FILAMENTS OF CELLU-
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This invention relates to a process for imparting the characteristics of wool to short filaments of cellulose derivatives such as cellulose esters and cellulose ethers, and more particularly it relates to a process for spinning threads or filaments of such derivatives which are too short to be easily spun, or to be spun at all, by the ordinary methods.

The treatment of fabrics, spun thread, or skeins of yarn for the purpose of imparting a wool-like appearance to these materials has been suggested. Thus, Patent 1,778,327 discloses the treatment of threads of spun filaments of cellulose acetate or fabrics containing them, with heated solutions of acids in order to give a dull appearance to the wool-like product made in this manner. Patent 1,765,581 discloses that treatment of cellulose acetate silk with certain salt solutions prevents the silk from losing its luster and becoming wool-like, thereby enabling textile material and fabrics containing this silk to be immersed without injury in hot liquors as is necessary in dyeing, etc. The present invention, which is concerned with the treatment of short lengths of filaments of cellulose derivatives, is to be sharply distinguished from those prior processes which are concerned merely with imparting a wool-like appearance to fabrics and continuous threads, or yarn, of cellulose derivatives rather than the production of a spinnable product from fibers too short to be easily spun. It is to be noted also that the temperatures of the solutions used in the mentioned patents are not sufficiently high for the optimum results in our improved process as hereinafter disclosed in connection with heated liquids. The boiling point of the highest boiling salt solution of the mentioned patent, 30% solution of calcium chloride, is 110.5° C. and the use of the above mentioned dilute acid solutions preclude a temperature sufficiently high to obtain the benefits of the invention disclosed herein.

We have discovered that the treatment disclosed herein of filaments of cellulose derivatives of short length (about 1½ to 6½ inches), known in the art as staple, produces such an intense crinkle in each of the filaments that the product has, not only a wool-like appearance, but has the characteristics of unspun wool itself in that it has the feel and other properties of wool and can be drawn out and spun like wool. An important part of our invention resides in our discovery that these short length filaments of cellulose derivatives can be converted into a wool substitute, and in our recognition of the fact

that these short fibers, previously of small commercial value, can, when sufficiently crinkled, be more readily spun into a more useful continuous thread or yarn than can the untreated fibers.

This invention has as an object a process for the preparation of a wool substitute. A further object is the manufacture of a continuous skein or yarn from short length filaments of cellulose derivatives. Other objects will appear hereinafter.

These objects are accomplished by the following invention in which short filaments of cellulose derivatives are treated in various fluid media, usually at elevated temperatures, in a loose condition wherein the ends of the fibers are free and the individual fibers free to contract so as to permit the desired crinkling to take place.

For the production of filaments of cellulose derivatives having more intense crinkle and a more wool-like appearance than can be produced by treatment with water or with low boiling point solutions we may use any one of several methods. An effective and economical method consists in heating the filaments in steam as more specifically set forth in the following two examples.

Example I

Filaments of dry cellulose acetate, cut into three inch lengths were heated in a steam chamber at 150° C. from boiler pressure for 30 seconds. Following this treatment cold water was run into the chamber to stop the reaction. The fibers were then dried and carded. The fibers so obtained were free from scroop, had an elasticity similar to wool, and had twenty to thirty crinkles per inch. The yarn obtained by spinning the fibers was soft and delustered.

Example II

Filaments of ethyl cellulose, cut into three inch lengths were heated in a steam chamber at 135° C. for thirty seconds. The crinkled fibers, after being dried and carded, were spun into yarn.

The operative range of temperature for the steam treatment of the cellulose acetate filaments varies somewhat from that of the ethyl cellulose filaments because of the lower melting point of the latter. The cellulose acetate filaments are crinkled between steam temperatures of 100° C. and 170° C. Sufficient crinkling takes place between 100° C. and 140° C. to render the filaments easily spinnable. The product obtained by steam treatment between 140° C. and 170° C. may be termed a wool substitute because the intense crinkling of the filaments at these temperatures

gives the product the characteristics of wool. The best operating range of time for temperatures between 100° C. and 140° C. is from about forty seconds to seventy seconds. Above 140° C. the time may vary from ten to sixty seconds, about thirty seconds being preferred. The ethyl cellulose filaments are crinkled in steam at temperatures between 100° C. and 140° C. Crinkling sufficient for spinning the filaments of ethyl cellulose into yarn takes place between 100° C and 125° C.; the crinkling between 125° C. and 140° C. being intense enough to impart wool-like characteristics to the product. The time of treatment for temperatures between 100° C. and 125° C. may vary from about forty seconds to seventy seconds. Above 125° C. the time may vary from ten seconds to sixty seconds, the best results being obtained with treatments of about thirty seconds' duration. In the case of either ethyl cellulose or cellulose acetate, it is undesirable to continue the heating longer than is necessary to give the maximum crinkling effect because deterioration takes place and the fiber strength is lowered rapidly. Results comparable to the crinkling resulting from the steam treatment previously set forth may also be obtained by heating the filaments of the cellulose derivatives in water and then drying quickly, and preferably in a current or blast of air. This combined effect of the water and air blast treatment is evidenced by the fact that the acetate or ethyl cellulose staple when treated one minute in water and dried rapidly in a loose condition in a blast or current of air is appreciably crinkled whereas samples of the same materials, when treated in water at 70° C. or at 100° C. followed by slow drying, were but slightly crinkled; in fact the product resulting from the water treatment at 100° C. with slow drying had fewer crinkles than the product resulting from the water treatment at 70° C. followed by rapid drying in an air blast. The following examples, 3 to 6, inclusive, indicate more specifically the preferred mode of practicing the invention by the combined water and air blast treatment.

Example III

A sample of two inch cellulose acetate staple filaments was treated one minute in water at the boiling point and then dried in a loose condition with a blast of air at room temperature. The crinkled fibers were then spun into yarn.

Example IV

Cellulose acetate filaments were cut into three inch lengths and treated with water at 80° C. for five minutes. They were then transferred to a glass chamber where they were heated with a blast of air, at 177° C. for five minutes. Twenty to thirty-five crinkles per inch were obtained. No charring occurred and the tensile strength was not decreased. The crinkled filaments were spun into yarn.

Example V

Cellulose acetate filaments were cut into three inch lengths; treated in water at 100° C., and dried in a glass chamber with a blast of air at 120° C. for five minutes. The fibers were then spun into yarn. The fibers contained twenty to twenty-five crinkles per inch. No charring occurred and the tensile strength was not decreased.

Example VI

A sample of ethyl cellulose filaments was treated one minute in water at the boiling point and then dried in a loose condition with a blast of air at room temperature. The crinkled fibers were then spun into yarn.

When using the combined water and air blast treatment referred to in Examples 3 to 6, the best results are obtained with water heated to temperature between 70° C. and the boiling point, the duration of the heat treatment being within the approximate range of sixty to one-hundred and twenty seconds. The temperature of the air blast may be at room or lower temperatures or at higher temperatures below the point at which the filaments are damaged. The water treatment prior to the air blast may be effected with aqueous solutions and it is to be understood, therefore, that the reference to an aqueous liquid in the claims is intended to comprehend water solutions as well as water alone.

The quick drying of the fibers by the air blast appears to be responsible, or partly responsible for the crinkling effect produced on the water treated fibers. While the quick drying with consequent crinkling can be effected more or less successfully by a very dry current of air of small velocity as compared to the blast referred to in the examples, we have found that the best results are obtained by using a blast of air strong enough to cause, and which causes agitation of the filaments during the air blast treatment. This superimposing of an air blast drying on the filaments previously treated with hot water produces a product that is almost as well crinkled as that resulting from a steam treatment within the temperature range favoring the best crinkling.

As indicated in Examples 3 to 6, it is preferred to use heated water before applying the air blast treatment. If the fibers are moistened with water below 70° C. and then subjected to a heated air blast, sufficient crinkles are formed to render the filaments easily spinnable as shown by the following example. The crinkling, however, is not sufficiently intense to produce a wool substitute.

Example VII

Cellulose acetate filaments cut into three inch lengths were moistened with water and then heated in a blast of air at 177° C. for five minutes. The filaments had nine to ten crinkles per inch. No charring occurred but the tensile strength decreased 5%.

Crinkling, lesser in degree than obtained in the preceding example, but sufficient to increase the spinning properties of the filaments may be effected by heating the filaments in a blast of heated air as shown by the following example:

Example VIII

Cellulose acetate filaments cut into three inch lengths were heated in a glass chamber with agitation by a blast of air at 177° C. for five minutes. Four or five crinkles per inch were obtained and no charring occurred.

Increased crinkling may be obtained by superimposing the air blast treatment on filaments previously crinkled by steam, provided that the maximum crinkling has not been effected by the steam treatment. Thus superimposing air blast drying on filaments previously crinkled by steam at the higher temperatures has no apparent effect. For instance, filaments of cellulose acetate when treated with steam between 150° C. and

160° C. and subjected to air blast drying, are apparently unaffected, but when the steam treatment is below 150° C., a subsequent air blast drying produces additional crinkling which increases with decreasing steam treatment temperatures. The superimposing of the air blast drying on ethyl cellulose previously crinkled with steam below 125° C. also produces an additional crinkling effect.

It is to be noted that no crinkling effect can be obtained by heating the filaments in an atmosphere of still air. Cellulose acetate filaments when heated in the presence of air in an oven, supported by a Monel metal screen between 100° C. and 180° C. at 10° C. intervals, did not crinkle and at 170° C. slight charring occurred. Beyond 170° C. the charring increases.

The crinkling of the filaments without deteriorating or weakening effects may also be accomplished by heating the filaments in liquids which have a sufficiently high boiling point and which prevent the escape of the naturally occurring moisture in the filaments. The following examples are illustrative of the effect of heating the filaments in glycerol.

Example IX

A sample of air dried cellulose acetate filaments, cut into three inch lengths, was heated thirty seconds in 95% water white glycerol at 165° C. After the filaments were removed, squeezed free from excess glycerol and washed in tap water at room temperature, they were dried at room temperature and spun into yarn. The crinkle was very intense, and the yarn was soft and of good quality.

Example X

The procedure of Example IX was carried out with like samples at 155° C. The crinkled fibers were carded and spun into yarn.

Example XI

The procedure of Example IX was carried out with like fibers at 140° C. The crinkled fibers were spun into yarn, but the crinkle was not so intense and the yarn not so soft as the yarn obtained by the heat treatment at 155° C. in the preceding example.

The treatment of the filaments of cellulose acetate in glycerol at 120° C. to 130° C. did not produce sufficient crinkling to make yarn. At a temperature of about 170° C. the filaments begin to fuse and the tensile strength is low. At 180° C., the filaments are fused completely and the fiber structure destroyed. The operative temperature range for treatment of cellulose acetate filaments in glycerol lies between about 140° C. and about 170° C. Temperatures between 140° C. and 155° C. render the fibers easily spinnable and the best crinkling effects are obtained between the approximate range of 155° C. to 165° C. The crinkling above 155° C. and below 170° C. is sufficiently intense to confer the properties of wool to the product. The reaction is complete within two minutes, but under certain conditions ten seconds is sufficient time. Ordinary water-white glycerol, containing about 5% water, is the most satisfactory although a dynamite grade of glycerol, containing about 1% water and of amber color, may be used without discolored the staple. Samples of ethyl cellulose filaments are crinkled in the same manner when treated with glycerol. The operative temperature range for the treatment of ethyl cellu-

lose filaments in glycerol is from 100° C. to 140° C. Above 140° C. the fibers are embrittled to a certain extent and at temperatures below 125° C. there is not so much crinkling. The range between 100° C. and 125° C. is sufficient to render these filaments spinnable, while the range from 125° C. to 140° C. produces an intensely crinkled product that is easily spun into yarn and also serves as wool substitute.

Among the various liquids that may be used to successfully crinkle filaments of cellulose derivatives, the following may be mentioned: Mineral oil, preferably in the form of the purified paraffin oil used for intestinal lubrication, cottonseed oil, coconut oil, lard oil, tallow, sperm oil, dicresyl carbonate and ethyl palmitate. Paraffin oil and cottonseed oil may be used for crinkling to almost the same extent as glycerine. Salt solutions are an effective crinkling medium and are of special importance because they are inexpensive. Examples of salt solutions are concentrated solutions of zinc chloride, magnesium sulphate, calcium chloride, etc. Solutions of sugar in water may also be used. With respect to all mediums, whether gaseous or liquid, it may be stated that the medium in which the filaments are heated to produce the crinkling effect must not carry away the naturally occurring moisture of the filaments and must not attack or dissolve the filaments at the temperature at which crinkling takes place. The enveloping blanket of gas or liquid must not decompose at the temperature at which crinkling of the filaments takes place. When the fibers are crinkled by heating in liquids, crinkling sufficient to enhance the spinning properties takes place when the liquid is heated within the range previously mentioned for the steam treatment. For more intense crinkling the liquid should be capable of being heated above 110.5° C., and preferably within the range of 125° C. to 170° C., the lower temperatures within this range being suitable for ethyl cellulose and the higher temperatures for cellulose acetate. Tricresyl phosphate, for instance, is not satisfactory because it dissolves the filaments of cellulose acetate and ethyl cellulose at the higher temperatures. Superheated water may be used but is not recommended because of the danger and difficulties involved. The higher temperatures may be used in any of the crinkling processes herein disclosed when it is essential to save time at the expense of a decrease in tensile strength. The advantages of using the lower temperatures for heating in water, steam or other medium are to be found in the retention of most of the original tensile strength of the products.

Filaments of cellulose derivatives other than those specifically mentioned, such as nitrocellulose and filaments of cellulose esters of other acids and of other cellulose ethers, may also be crinkled in accordance with the process of the present invention. Among the filaments of cellulose esters may be mentioned cellulose laurate, cellulose phthalate, cellulose formate, cellulose propionate and cellulose butyrate. Examples of other cellulose ethers that may be made into filaments and crinkled by the process of the present invention are benzyl cellulose, propyl cellulose, butyl cellulose and methyl cellulose. These cellulose ethers are such as are formed when an alkali cellulose reacts with the hydrocarbon chloride.

The filaments of cellulose derivatives and especially the esters and ethers when crinkled by the present process under conditions which produce the more intense crinkling have a dull finish,

very little or no scroop and resemble fine wool in appearance. The springiness, elasticity and drawing properties are similar to wool fibers. The crinkled filaments are also similar to wool fibers in feel and in heat insulating value. The products can be used for most purposes for which wool is used. The filaments can be made into finer deniers than wool fibers occur and they are more uniform in size and properties than wool.

- 10 The more intensely crinkled wool-like fibers are more readily spun into yarn than the fibers that have been crinkled to a lesser degree by the milder heat treatments. The spinning properties of the latter fibers are, however, considerably enhanced as compared to the untreated filaments. The practice of the process of the present invention is economical because of the low material cost and simplicity of operation.

- The crinkled filaments may be drawn out and spun into yarn for cloth, and this capability of being spun enhances the value of the staple filaments which otherwise are less desired and less useful than the long continuous filaments. An important feature of the invention resides in the treatment of the short staple fibers as herein disclosed and the spinning of these short filaments into yarn. The short filaments because of their loose ends take a more intense crinkle than a continuous thread. Furthermore it is not satisfactory to treat the filaments when in skein form, or in lengths appreciably greater than 6½ inches. The filaments must be loose and free to move and cannot be held taut or wound tightly in place during treatment. We have found that the treatment of the filaments in long lengths results in such tangling as to make the process difficultly practical inasmuch as the tangled threads cannot be pulled apart as can the shorter filaments.

- As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that we do not limit ourselves to the specific embodiments thereof except as defined in the appended claims.

- 45 We claim:

1. A process of manufacturing a wool substitute which comprises crinkling loose unspun filaments of cellulose acetate having a length from about 1½ inches to 6½ inches by heating such filaments in steam under pressure within the temperature range of 140° C. to 170° C.

2. A process of manufacturing a wool substitute which comprises crinkling loose unspun filaments of ethyl cellulose having a length from about 1½ inches to 6½ inches by heating such filaments in steam under pressure within the temperature range of 125° C. to 140° C.

3. A process which comprises treating loose unspun filaments of cellulose derivatives having a length from about 1½ inches to 6½ inches by heating such filaments in a fluid which retains moisture in the filaments, removing the filaments from the fluid, and drying the filaments by agitating them in a loose unspun condition with a blast of air, whereby said filaments are crinkled and thereby made spinnable.

4. A process which comprises treating loose unspun filaments of cellulose derivatives having a length from about 1½ inches to 6½ inches by heating such filaments with steam and drying the filaments by agitating them in a loose unspun condition with an air blast whereby said filaments are crinkled and thereby made spinnable.

5. A process which comprises treating 1½ to 6½ inch length filaments of cellulose derivatives selected from the class consisting of cellulose acetate filaments and ethyl cellulose filaments in a loose unspun condition with water within the temperature range of 70° C. and the boiling point, and drying the filaments by agitating them in a loose unspun condition with an air blast, whereby said filaments are crinkled and thereby made spinnable.

6. A process of manufacturing yarn from filaments of cellulose acetate too short to be readily spun which comprises crinkling such filaments by heating them while in a loose unspun condition within the temperature range of 140° C. to 170° C. in a liquid which retains the moisture in the filaments and spinning the crinkled filaments into yarn.

7. A process of manufacturing yarn from filaments of ethyl cellulose too short to be readily spun which comprises crinkling such filaments by heating them while in a loose unspun condition within the temperature range of 125° C. to 140° C. in a liquid which retains the moisture in the filaments and spinning the crinkled filaments into yarn.

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