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PROCESS FOR INCREASING THE CRYSTALLINITY AND SAFE IRONING TEMPERATURE OF CELLULOSE TRIACETATE TEXTILES WITH ACETONE MIXTURES

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This application relates to textile materials comprising fibres of cellulose triacetate, and is a continuation in part of Application Ser. No. 467,223 filed November 5, 1954.

In U.S. Application Ser. No. 400,798, filed December 28, 1953, now U.S. Patent No. 2,862,785, there is described a type of cellulose triacetate textile material which is characterized inter alia by having a sticking temperature in the neighbourhood of 220°—240° C., as compared with the sticking temperature of about 200° C. of cellulose triacetate materials as previously made and of materials made from the more usual acetone-soluble type of cellulose acetate. (The sticking temperature is a measure of the sensitivity of the material to temperature, and for the purpose of the present specification is to be taken as the lowest temperature at which a heated iron, after standing on a fabric of the cellulose acetate for 10 seconds, causes the fabric to stick to its surface.) These new materials are obtained by subjecting cellulose triacetate materials to the action of dry heat at temperatures above about 170° C. or to the action of a heated softening agent such as steam or hot water at a temperature between about 120° and 160° C. This treatment changes the physical form of the cellulose triacetate (although not, at least to any significant extent, its chemical composition). Examination of the X-ray diagrams of the cellulose triacetate fibres before and after the treatment has confirmed that the change is accompanied by a rearrangement into a state of greater order of the macromolecules of the cellulose triacetate, or in other words an increase in the proportion of the triacetate which is in a more or less crystalline state and a decrease in the proportion which is in the so-called amorphous or substantially non-crystalline state. For the purpose of the present specification it will be convenient to refer to the new materials as materials of cellulose triacetate "of an increased degree of crystallinity"; similarly cellulose triacetate materials as first obtained by melt-spinning or wet- or dry-spinning processes, or by completely acetylating unoriented partly acetylated cellulose filamentary materials, will be termed materials of cellulose triacetate "of a normal degree of crystallinity."

Textile materials comprising fibres of cellulose triacetate of an increased degree of crystallinity have a number of advantages. One such advantage, which is of considerable commercial value, is their high safe ironing temperature. It is well known that while cellulose materials such as cotton, linen and regenerated cellulose fabrics can be ironed at temperatures up to about 240° C., at which temperature they begin to scorch, the cellulose acetate fabrics obtained in the past have been more sensitive; indeed in practice it is recommended that ironing temperatures above 170°–180° C. should not be used, since unless the operator is very skilful they tend to impart a gloss or shine to the fibres. This temperature of 170°–180° C. is commonly referred to as the safe

ironing temperature. Other thermoplastic fibres, including nylon, have similar or lower safe ironing temperatures.

According to the invention, textile materials comprising fibres of cellulose triacetate of a normal degree of crystallinity are given a treatment in which the degree of crystallinity of the cellulose triacetate is increased by the action of a mixture of acetone with water or a lower monohydric alcohol (i.e. a monohydric alcohol containing up to 4 carbon atoms in the molecule) which mixture is a swelling agent for cellulose triacetate at 15° C. but contains at most 70% of acetone. An important aspect of the invention is the manufacture of textile materials made up at least in part of thermoplastic fibres and having a safe ironing temperature substantially higher than 180° C., and especially between 195° and 230° C., which comprises subjecting a textile material comprising as the sole thermoplastic constituent fibres of cellulose triacetate of a normal degree of crystallinity to a treatment in which the degree of crystallinity of the cellulose triacetate is increased, and the sticking temperature of the material is raised by at least 10° C., by the action of the aqueous or alcoholic acetone. Preferably an aqueous acetone containing 30–70% by weight of acetone, or an alcoholic acetone containing 20–70% by weight of acetone, is used.

The term "Textile material" is employed in this specification to denote both textile fabrics (and articles made from them) and intermediate structures used in making fabrics, such as yarns made up of continuous filaments or spun from staple fibres, and staple fibres themselves in the form of tops, slivers and other structures in which they are more or less loosely held together. The term "Fibres" includes both continuous filaments of indefinite length and short lengths of filament such as staple fibres.

Cellulose triacetate textile materials of a normal degree of crystallinity which are to be treated in accordance with the invention may with advantage be formed by a melt spinning process, especially by a process described in U.S. Applications Ser. Nos. 243,994, filed August 29, 1951; 292,771, filed July 10, 1952; 292,772, filed July 10, 1952 now U.S. Patent No. 2,888,711; 304,441, filed August 14, 1952; 338,834, filed February 25, 1953 now U.S. Patent No. 2,831,748, or 423,843, filed April 16, 1954 now U.S. Patent No. 2,742,469. In one such process, cellulose triacetate in powder form is urged (e.g. by a rapidly reciprocating tamper) against the side of a heated plate having spinning orifices therein and is fused by the heat applied to the plate, the fused ester is drawn away from the orifices in the form of filaments, and fresh powdered ester is continuously fed to the plate. The spinning orifices may be circular, or they may be in the form of slits or of two or more closely spaced or intersecting holes, as described in U.S. application Ser. No. 338,834. In another process cellulose triacetate in the form of a coherent rod or a block or tablet of uniform cross-section is pressed axially against a heated plate having spinning orifices therein. The cellulose triacetate may contain pigments, dyes or other effect materials, as described for example in U.S. application Ser. No. 292,771. Wet and dry spinning methods may also be used to form the cellulose triacetate textile material. Thus solutions of cellulose triacetate in mixtures of methylene or ethylene chloride and methyl or ethyl alcohol or acetic acid, or in acetic acid alone, may be extruded as filaments and set either by means of a coagulat-

ing liquid or by an evaporative method. For example solutions of cellulose triacetate in mixtures of methylene or ethylene chloride and acetic acid may be extruded into a coagulating liquid comprising an aqueous alcohol, especially aqueous ethyl alcohol of concentration about 90–95% as described in U.S. Patent No. 2,657,973, or solutions in acetic acid may be extruded into aqueous acetic acid.

Yet another method by which the cellulose triacetate textile material may be obtained is the further acetylation of a textile material comprising unorientated yarns of cellulose acetate of a lower acetyl value. For instance, yarns or fabrics of acetone-soluble cellulose acetate may be further acetylated with acetic anhydride in the presence of a diluent such as benzene, and of a basic or acid esterification catalyst such as pyridine, sulphuric acid, or hydrochloric acid with or without a metal chloride such as zinc chloride or ferric chloride as described in British Patents Nos. 448,816, 448,817 and 448,917.

It is an advantage of the invention that the treatment does not require the use of high temperatures. Thus the textile material can be treated with the mixtures of acetone and water or a lower monohydric alcohol at temperatures down to about 15° C., in particular temperatures between 15° and 40° C. Another advantage is the short time necessary; thus a very useful effect can be obtained in half an hour or less, e.g. in 3–30 minutes, though longer treatments can be applied if desired. For example the textile material may be immersed in a bath of the aqueous or alcoholic acetone for substantially the whole of the desired treatment time, and then squeezed or centrifuged and dried quickly, or it may be immersed for a short time and then allowed to stand before it is squeezed or centrifuged and dried or it may be squeezed or centrifuged at once and then allowed to dry relatively slowly.

In the course of the treatment the textile materials tend to shrink somewhat. This shrinking may be prevented by keeping the material under tension, or the material may be allowed to shrink either freely or by less than it would in the complete absence of tension.

The treatment may be applied either to individual batches of materials, e.g. lengths of fabric or loosely wound yarn packages which will permit shrinking, or continuously as to a running fabric or yarn. Yarns and the like are usually best treated by a continuous method owing to the difficulty of allowing for uniform shrinkage if they are in package form. For example, they may be treated on thread-storage thread-advancing reels and like devices arranged so as to permit the desired degree of shrinkage.

Besides the high safe ironing temperature already referred to, materials of cellulose triacetate of an increased degree of crystallinity also have very good dimensional stability at high temperature. Thus they may in many cases be heated to 220° to 235° C. without undergoing much change in dimensions, whereas cellulose triacetate materials which have not been treated either in accordance with the present invention or by a process such as those described in U.S. applications Ser. Nos. 400,798 and 467,223 and also materials made from acetone-soluble cellulose acetate, on being heated to such temperatures undergo a very considerable degree of shrinking and also suffer a serious loss of strength. Again, the tendency of cellulose triacetate textile materials to "creep" under load in hot water is greatly reduced by increasing the degree of crystallinity of the cellulose triacetate.

The invention is illustrated by the following examples:

Example 1

A fabric woven from a melt-spun cellulose triacetate yarn obtained by the process of U.S. application Ser. No. 243,994 was treated for 20 minutes with a mixture of equal parts by weight of acetone and water at about 20° C., with the result that its safe ironing temperature was raised from about 170° to 210° C. Treatment under the same conditions with a mixture of 40% by weight of acetone and 60% by weight of ethyl alcohol had a similar effect.

Example 2

A fabric woven from a mixture yarn spun from 50% cellulose triacetate staple fibre and 50% cotton, and having a safe ironing temperature of about 170° C., was immersed for five minutes in 65% aqueous acetone at 20° C., squeezed and dried. Its safe ironing temperature was raised by this treatment to about 210° C.

In both these examples the effect of the treatment has been illustrated by the rise in the safe ironing temperature of the fabric, since this is a property of immediate practical importance. This rise was, of course, always accompanied by a rise in the sticking temperature and an increase in the dimensional stability of the fabric. Thus the treated fabrics could be heated to about 235° C. for 1 minute without undergoing any substantial shrinkage, whereas the untreated fabrics when heated to this temperature shrank by 20 to 25%. Furthermore the tendency of the untreated all-cellulose triacetate fabric to creep under any specified load in water at 80° C. was reduced by up to 75–80%.

The treatment of the present invention has moreover a further advantage, which is not shared by the processes specifically described in application Ser. No. 400,798 or now claimed in application Ser. No. 467,223, namely that simultaneously with its valuable effect on the safe ironing temperature and dimensional stability of the cellulose triacetate materials, it also causes a worth-while increase in their voluminosity and yarn abrasion resistance. This is shown by the following example.

Example 3

A cellulose triacetate yarn made by dry spinning from solution in a mixture of methylene chloride with a small proportion of methanol had a voluminosity of 1.4 (on an arbitrary scale) and an average abrasion resistance (measured by a loop on loop method) of 1105 cycles to break. The yarn was immersed in 65% aqueous acetone for 1 minute at about 20° C. and was then padded and dried at the same temperature. The safe ironing temperature of the material was raised by about 35° C., its voluminosity was 2.2 and its average abrasion resistance 2409 cycles to break. Further experiments in which a fabric woven from the yarn was treated with the aqueous acetone and then tested for flex abrasion resistance and also by the ball penetration method showed that the treatment is unique, so far as is known, in causing an important increase in the resistance of the fabric to both these forms of abrasion.

Having described our invention what we desire to secure by Letters Patent is:

1. A process for the manufacture of textile materials containing cellulose triacetate fibres which comprises bringing a textile material comprising as the sole thermoplastic constituent fibres of cellulose triacetate which is in that crystalline state in which fibres thereof have a sticking temperature about 200° C. into contact with a mixture at a temperature of 15° to 40° C. consisting of from 30 to 70 percent by weight of acetone and from 70 to 30 percent by weight of a compound selected from the group consisting of water and monohydric alkanols containing up to 4 carbon atoms in the molecule and maintaining the textile material in contact with the said mixture in the temperature range of 15 to 40° C. until the safe ironing temperature of the said cellulose triacetate fibres is raised to between 195° C. and 230° C.

2. A process as claimed in claim 1 wherein the mixture used is a mixture of acetone and water.

3. A process as claimed in claim 1 wherein the mixture used is a mixture of acetone and methyl alcohol.

4. A process as claimed in claim 1 wherein the tex-

tile material is in the form of an intermediate structure used in making fabric.

5. A process as claimed in claim 4 wherein the textile material is immersed in said mixture.

6. Process according to claim 1, wherein the textile material consists entirely of the cellulose triacetate fibres.

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