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TREATMENT OF FIBERS OR LIKE MATERIALS

Henry Dreyfus, London, England

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8 Claims. (Cl. 8—20)

This invention relates to improvements in the treatment of fibers or like materials and is particularly concerned with the manufacture of filaments, threads, yarns, ribbons and the like of cellulose acetate or other cellulose esters or ethers.

In U. S. Patent No. 1,709,470 processes are described for the treatment of cellulose acetate filaments, threads or the like in which such products are subjected to a stretching operation at any stage of their manufacture after they have reached the fully set and finished condition, such stretch being beyond the elastic limit, the products thereby acquiring an increased resistance to the action of hot or boiling aqueous media, and also being reduced in denier. Such a stretching results in an increased tensile strength. U. S. application S. No. 378,684 filed 16th July, 1929 describes how the stretching of such filaments, threads, yarns or the like of cellulose acetate or other cellulose esters or ethers may be effected in a continuous operation, that is to say while the materials are travelling from point to point, for example from bobbin to bobbin or from any other package to a winding device or from a dry spinning machine to a winding device. The permanent stretch may be as much as 100% or more of the original length of the filaments or other materials. The stretching is assisted by treating the materials either before or during the stretching operation with solvents or softening agents for the cellulose acetate or other cellulose derivative.

Again, U. S. applications S. Nos. 402,785 filed 20th October, 1929, 418,414 filed 3rd January, 1930, 437,423 filed 20th March, 1930, 589,428 filed 28th January, 1932, 681,043 filed 24th March, 1932 and 607,668 filed 26th April, 1932 describe processes in which solutions of cellulose acetate or other cellulose esters or ethers are spun by wet spinning methods into coagulating media containing high concentrations of solvents or other softening agents for the materials. By this means the materials are obtained in a plastic state in which they may be stretched to a considerable extent.

I have now found that improved results, particularly as regards tenacity, are obtained by including with the solvent or other softening agent an oily or a fatty body or a soap. The oil, fat or soap may be soluble in the medium used to assist the stretching operation or it may be insoluble therein, in which case it should be emulsified in the medium. Any suitable animal or vegetable oil or a soap which may be made from an animal or vegetable oil, including sulphonated soaps, may be used for the purpose of the present invention. Thus, for instance, there may be used castor oil, olive oil, cocoanut oil, arachis oil, cotton seed oil, rape oil and whale oil, raw or hardened. Again, there may be employed the soaps obtainable from such oils, for example sodium, ammonium or potassium oleate or ethanolamine oleates or other organic soaps of oleic acid, the soaps obtainable from the cocoanut oil fat acids, and the soaps of lauric, palmitic, stearic or ricinoleic acids or sulphonated soaps obtainable from any of these compounds. Other fatty bodies, such as sulphonated fatty alcohols and the ω-alkylamides of fatty acids and their sulphonated derivatives, may be used for the purposes of the present invention, while examples of aromatic compounds are the alkylated naphthalene sulphonic acids, e. g. isopropyl naphthalene sulphonic acid. Most of the above substances are wetting agents, and it is found that the use of this class of substance broadly is of value for the purpose of the present invention.

In general proportions of 1–5 up to 10% of oil or the like on the total softening medium are suitable, though the invention is not limited thereto, and lower or higher proportions may of course be employed.

In most cases the oils will be soluble in the medium used to assist the stretching treatment. If insoluble they may be emulsified therewith, for example by means of soaps, sulphonated fats or oils or salts thereof, such as sulphonated castor oil, true sulphonic acids of aliphatic hydrocarbons or alcohols or of fatty acids or salts thereof, ethanolamine and other alkylolamine soaps and amino-alkylamides of higher fatty acids and in the aromatic series naphthalene sulphonic acid compounds and particularly alkylated naphthalene sulphonic acids, such as propyl and iso-butyl naphthalene sulphonic acids and the sulphonated condensation products of formaldehyde and naphthalene. Such emulsifying agents are particularly useful where the swelling medium is aqueous in character. Where the swelling medium consists of a solvent or strong swelling agent for the cellulose derivative and a non-aqueous diluent the oil or fat will be soluble therein in practically all cases. Magnesium or even calcium soaps are suitable emulsifying agents where the continuous phase of the emulsion required is non-aqueous.

As examples of suitable solvents or swelling agents for assisting the stretching of the cellulose acetate or other cellulose ester or ether the fol-

lowing may be instanced:—aqueous solutions of thiocyanates, such as those of the alkalies, ammonium and alkaline earths, aqueous solutions of zinc chloride, acetic acid, formic acid, lactic acid, diacetone alcohol, acetone and the like; the ethers, esters or ether-esters of olefine glycols and polyolefine glycols or of other polyhydric alcohols for instance the mono- and di-methyl and ethyl ethers of ethylene glycol, the mono-methyl and ethyl ethers of propylene glycol, and the monoethyl ether of diethylene glycol, dioxane, methylene ethylene dioxide and their homologues and substitution products, e. g. dioxane dicarbinyl diacetate, glycol mono-acetate, methyl glycol monoacetate, mono-, di-, and tri-acetins, ethyl lactate etc.; phenols, formaldehyde acetaldehyde, dichlorethylene methylene chloride and the like, or mixtures of these, or solvent mixtures. In most cases such solvents or swelling agents may if necessary be diluted with water, though the methylene chloride or dichlorethylene type of solvent is immiscible with water, so that dilution should be effected with a non-aqueous diluent miscible therewith, as for example benezene, carbon tetrachloride, perchlorethylene and the like.

The stretching operation may in the present invention be conducted on the travelling filaments, yarns, etc., as described in U. S. application S. No. 378,684, the operation being conducted for example in a bobbin to bobbin operation, or in a cheese winding operation or in any other rewinding operation or in the course of the travel of the filaments from a wet or dry spinning machine to a winding device. As is indicated in U. S. application S. No. 662,844 filed 2nd April, 1932, considerable advantages accrue by carrying out the stretching operation while the yarns, threads or the like are in warp formation, that is to say a large number of threads are aligned side by side and are stretched by a single stretching device. The stretch may be inserted in one single stretching operation or it may be inserted in stages, in which case it is of advantage as described in U. S. application S. No. 573,424 filed 6th November, 1931, to maintain the filaments or the like in a softened or plastic state between the stretching stages.

Prior to the softening treatment which immediately precedes the stretching or which is effected simultaneously therewith, the materials may be subjected to a pretreatment with softening agents of strengths different from that used in the final softening treatment, as is described in U. S. application S. No. 638,776 filed 20th October, 1932. The process of the present invention may be employed in connection with any one or more of such softening treatments.

Instead of carrying out the process of the present invention on the materials during their travel from one point to another, they may if desired, be treated in hank or other package form.

The amount of stretch inserted in the threads in the present invention may vary from comparatively small stretches, such as 20 to 25% to 50 or 100 or 200% up to 500% or more of the original length of the filaments.

The process of the present invention may be applied to the treatment of filaments, threads, yarns or other materials made of or containing cellulose acetate or other cellulose esters or ethers of all kinds, for instance cellulose formate, propionate, butyrate, nitro-acetate, or mixed cellulose esters and cellulose esters including higher fatty acid radicles, for instance the laurate radicle, or cellulose ethers, for instance ethyl or benzyl celluloses, or unsaturated cellulose ethers or mixed ethers, for instance the mixed ethyl benzyl cellulose or mixed ether-esters of cellulose, e. g. ethyl cellulose acetate and oxyethyl cellulose acetate. The esters or ethers may be of low, medium or high viscosity characteristics; for instance cellulose acetates having a viscosity of 5–10, 20–30 or 50–100 or 200 or more as measured by comparing the viscosity of a 6% solution in acetone against a standard of glycerine taken as 100 may be present in the materials. The yarns or other materials may consist wholly of the cellulose ester or ether or may contain other materials not deleteriously affected by the treatment.

Continuously with the stretching according to the present invention cellulose ester materials may be subjected to a saponification which may be superficial, may be partial and uniform or may be complete. Such saponification may take place immediately after the stretching treatment of the present invention, or, particularly where the cellulose esters are subjected to a partial and substantially uniform saponification, may immediately precede the stretching treatment. The saponification may be effected with the aid of aqueous or aqueous alcoholic or alcoholic caustic soda, caustic potash, trisodium phosphate or other alkaline salt or reagent or may be effected with ammonia or organic amine in the vapour state. The saponifying media may contain sodium acetate or soaps or other buffer substances and may contain in addition to or in place of methyl alcohol or ethyl alcohol lime or other substances whereby the amount of caustic soda necessary for effecting any desired degree of saponification may be less than that theoretically necessary to produce that saponification. Such processes are described in U. S. application S. No. 655,778 filed February 8, 1933. As described in U. S. application S. No. 668,068 filed on even date herewith the saponifying media may themselves contain oils or fats as with the stretching media of the present invention.

The following examples are given in order to illustrate the invention but it is to be clearly understood that they do not limit it in any way:—

Example 1

A cellulose acetate yarn in hank form is softened in a bath consisting of an emulsion of linseed oil in aqueous ethyl lactate prepared e. g. in the following manner. 8 parts of oleic acid, 2 parts of tri-ethanolamine and 30 parts of linseed oil are thoroughly mixed at ordinary temperatures and 30 parts of 50% aqueous ethyl lactate are then slowly added to the mixture with constant and vigorous stirring until an emulsion is obtained. A further 60 parts of linseed oil are then added in small proportions with continual stirring. The emulsion is then diluted to 10 times its volume with 50% ethyl lactate which is added in small proportions at the beginning with constant stirring and then in larger amounts until the whole has been introduced and a satisfactory emulsion is obtained.

The hanks are soaked in the emulsion for about 5–10 minutes at 20–25° C. and then stretched, for example by 100 to 200% of their original length and are found to have an increased tenacity.

Example 2

A number of cellulose acetate threads are taken from a creel of bobbins and led over a roller in warp formation into a stretching bath consisting

of a 45–50% solution of dioxane containing 1 to 2% of the sodium salt of the sulphuric ester of lauryl alcohol. From the feed roller the warp passes through a reed to keep the threads apart, under a guide and then down the bath which is 80 to 100 feet long, through a reed at the further end, round a draw-roller and then to washing, drying and winding devices. The peripheral speed of the feed roller is about 5 to 7 metres per minute while that of the draw-roller is 20 to 30 metres per minute so that a stretch of 400 to 500% is obtained. The temperature of the bath is 20 to 25° C. The threads which have been stretched according to the example are found to have considerably greater tenacity than before stretching.

Example 3

A 25% solution of cellulose acetate in acetone is extruded into a coagulating bath consisting of a 50 to 55% solution of diacetone alcohol and containing from 1 to 2% of the sodium salt of isopropyl naphthalene sulphonic acid. The filaments pass through the bath for a distance of 10 to 12 inches and then round a roller rotating at a peripheral speed of 40 to 50 metres per minute so that they are drawn down to low denier, for example 1 to 1.5. After passing over a draw-roller the filaments are washed to remove excess solvent, preferably with solutions of solvent in lower concentration or with salt solutions and are then dried and wound. Materials having an improved tenacity are thus obtained.

The term "swelling agent" as employed hereinafter in the claims is to be understood as including not only agents which exert only a swelling action on the cellulose derivative, but also agents which are true solvents therefor.

What I claim and desire to secure by Letters Patent is:—

1. In a process for softening and stretching artificial filaments, threads, yarns, ribbons and like materials containing organic derivatives of cellulose, the steps of softening the materials with a medium throughout which are uniformly distributed a swelling agent for the derivative of cellulose and a substance selected from the group consisting of oils, fats and soaps and stretching the materials.

2. In a process for softening and stretching artificial filaments, threads, yarns, ribbons and like materials containing cellulose acetate, the steps of softening the fully-formed materials with an aqueous medium throughout which are uniformly distributed a swelling agent for cellulose acetate and a substance selected from the group consisting of oils, fats and soaps and stretching the materials.

3. Process for the manufacture of high tenacity filaments, threads, yarns, ribbons and like materials containing cellulose acetate, which comprises forming the materials by extruding a suitable solution of cellulose acetate into an aqueous medium throughout which are uniformly distributed a swelling agent for cellulose acetate and a substance selected from the group consisting of oils, fats and soaps, and stretching the materials during their formation.

4. In a process for softening and stretching artificial filaments, threads, yarns, ribbons and like materials containing organic derivatives of cellulose, the steps of softening the fully-formed materials with an aqueous medium in which are dissolved a swelling agent for the derivative of cellulose and a substance selected from the group consisting of oils, fats and soaps, and stretching the materials.

5. Process for the manufacture of high tenacity filaments, threads, yarns, ribbons and like materials containing organic derivatives of cellulose, which comprises forming the materials by extruding a suitable solution of the cellulose derivative into an aqueous medium in which are dissolved a swelling agent for the cellulose derivative and a substance selected from the group consisting of oils, fats and soaps, and stretching the materials during their formation.

6. In a process for softening and stretching artificial filaments, threads, yarns, ribbons and like materials containing cellulose acetate, the steps of softening the fully-formed materials with an aqueous medium in which are dissolved a swelling agent for cellulose acetate and a substance selected from the group consisting of oils, fats and soaps, and stretching the materials.

7. Process for the manufacture of high tenacity filaments, threads, yarns, ribbons and like materials containing cellulose acetate, which comprises forming the materials by extruding a suitable solution of cellulose acetate into an aqueous medium in which are dissolved a swelling agent for cellulose acetate and a substance selected from the group consisting of oils, fats and soaps, and stretching the materials during their formation.

8. In a process for softening and stretching artificial filaments, threads, yarns, ribbons and like materials containing cellulose acetate, the steps of softening the fully-formed materials with an aqueous medium in which are dissolved dioxane and a substance selected from the group consisting of oils, fats and soaps, and stretching the materials.

HENRY DREYFUS.