Patented Feb. 8, 1949 2,461,043

UNITED STATES PATENT OFFICE

2,461,043

PROCESS OF CONDITIONING CELLULOSE ESTER FILAMENTS

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No Drawing. Application November 10, 1944, Serial No. 562,908

3 Claims. (Cl. 117—139.5)

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This invention is concerned with novel conditioning materials which soften and lubricate filamentary materials, such as yarns, fibers, or fabrics of organic derivatives of cellulose, such as cellulose esters or ethers including cellulose acetate, ethyl cellulose, cellulose aceto-propionate and cellulose aceto-butyrate, and is a continuation in part of my earlier application Serial No. 470,664, filed December 30, 1942, now abandoned.

The application of conditioning agents to artificial textile materials of the nature specified above is a common expedient to prepare the materials for textile processing operations or to impart a desired "hand" in the final product. For example, the conditioning agents may be applied to soften and lubricate textile staple fibers to facilitate their conversion by such processes as carding, picking, opening, and spinning into an intermediate product known as spun rayon yarns. Such yarns may then be converted by weaving, knitting, braiding or related processes into a final fabric. Alternatively, the conditioning agents may be applied to continuous filamentary yarns of the organic derivative of cellulose to facilitate their conversion into textile fabrics. Again, fabrics themselves after their formation by weaving, knitting, and the like may be treated with the conditioning agent to impart an improved softness of hand thereto.

However, in the manufacture of cellulose acetate continuous filament yarns by the dry-spinning process, it is necessary to apply such conditioning materials shortly after the formation of the filaments but before their first collection, which is generally accomplished by some form of twisting device, such as a cap or ring twister. The point of application to the travelling untwisted bundle of filaments may be within the spinning cell between the spinneret and the first guide, or it may be along the course of the bundle between this guide and the draw roll or godet where the bundle undergoes a certain amount of stretching due to the high speed of the draw roll. A number of exacting conditions must be fulfilled in order that a conditioning material can be used satisfactorily in this situation. First of all, it must be capable of rendering the filaments soft as contrasted with "harsh." Secondly, it must lubricate the filaments. It must be present on the finished yarn exclusively in the form of a substantially continuous liquid phase regardless of the number of ingredients of which it may be composed even in the presence of any residual spinning solvent contained in the yarn at the time of application, and it must remain

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in the liquid condition throughout any period of storage under all temperatures from about 0° C. to 25° C. and higher. Otherwise, the yarn develops too much friction and static to be collected satisfactorily on modern high speed winding and/or twisting equipment. It must not require any substantial amount of water or volatile solvents to render it a homogeneous liquid under these ordinary room temperature conditions even though it may comprise several ingredients; otherwise the yarn becomes "overstretched" and has abnormally low extensibility, or it becomes impossible to spin the filaments at all, especially when operating at high delivery speeds. Still further the conditioning material should reduce the tendency of the filaments to build up electrostatic charges by rubbing action upon guides and other relatively moved parts during textile fabrication, such as weaving and the like.

It is extremely rare that all of these capabilities can be found sufficiently pronounced in a single substance, and it is difficult to find two or more different substances which can be compatibly blended, whose capacities can complement each other to attain all the qualities needed in a conditioning material enumerated above.

It has now been discovered that an entirely new class of compounds not heretofore used for this purpose has the ability to soften and lubricate organic derivatives of cellulose, which may be at the time of application in the form of fibers, continuous filaments, spun or continuous filament yarns (and especially freshly formed yarns during dry spinning) or textile fabrics, and that their use as conditioning agents is characterized by marked incidental advantages not commonly found associated together with any particular group of compounds. This group of compounds may be generally designated as the inner ethers or inner anhydrides of hexitols which are partially esterified with a higher fatty acid of at least 8 carbon atoms and the derivatives of such partial esters obtained therefrom by condensing them with ethylene oxide. The partial esters of the inner ethers or anhydrides of such hexitols as mannitol, sorbitol, iditol, and dulcitol from such acids as stearic, oleic, ricinoleic, palmitic, myristic, lauric, or from mixed fatty acids, such as are obtainable from fish oils, vegetable oils, or animal fats which may or may not be hydrogenated, are applicable. Preferably, such partial esters also contain ethylene oxide residues obtained by the reaction of the partial ester of the inner ether with ethylene oxide or by the

condensation of the inner ether with ethylene oxide prior to esterification. The content of the ethylene oxide may vary within wide limits from one to twenty or more ethylene oxide units per molecule of the inner ether or of the partial ester thereof. Depending upon the properties desired for the particular application of the conditioning agent, the control over the content of ethylene oxide units in the molecule can serve to balance the content of fatty acid radicals with a hydrophilic content to any desired point. In general, the greater the ethylene oxide content, the less tendency there is for the development of electrostatic charges on the treated yarns. However, the introduction of too great a number of ethylene oxide units in the molecule tends to decrease the lubricity so that it is preferable to make a compromise to obtain the optimum results. For example, a compound containing a large proportion of fatty acid radicals, such as sorbitan trioleate, may contain a relatively large proportion of ethylene oxide units in the molecule (e. g. twenty) to balance the hydrophobic character of the three oleyl radicals for adapting the compound to use as a conditioning agent, and this relationship is also advantageous when it is desired to obtain rapid and complete removal of the conditioning agent subsequent to the textile processing operation for which it is applied. On the other hand a compound such as sorbitan mono-oleate when applied for the same service would need relatively few ethylene oxide units (two or three) or none to produce the optimum of softening, lubricity and antistatic qualities and to assure its rapid and complete removal subsequently.

These compounds in the form of their technical grades available on the market vary from liquid to pasty wax-like masses, depending mainly upon the degree of saturation of the fatty acid chains and to some extent upon the length of such chains. For example, the unsaturated esters, such as the oleates, are liquids while the palmitates, myristates, stearates, and laurates are pasty wax-like masses. Those that are liquids can be applied directly as such without compounding. However, the others have the capability of being readily dispersed in non-volatile liquid media, such as mineral oil, to form a substantially continuous liquid phase of excellent stability at ordinary room temperatures of about 15° to 25° C. encountered during storage as well as at the higher temperatures (e. g. up to about 45° C.) normally encountered in dry-spinning rooms in which such conditioning materials are frequently applied. The fact that the compounds are themselves liquids or form in mineral oil liquid conditioning media facilitates their application at normal room temperature and especially assures high fluid consistencies at spinning room temperature. It has been found that the conditioning media exhibit a high degree of lubricity under all usual conditions regardless of humidity.

The dual hydrophobic-hydrophilic character of these compounds, which, as stated above, may be varied within a wide range by balancing or by varying between opposite extremes the fatty acid and ethylene oxide content, is apparently accountable for some of their main advantages. Their non-volatility assures permanency and stability of their conditioning effect upon textile materials to which they are applied, under most conditions of humidity, temperature, and exposure to light. At the same time, this dual character apparently accounts for the high degree of lubricity of the textile materials treated therewith. In addition, in spite of the pronounced hydrophobic portion of the compound, their hydrophilic nature is sufficient to assure efficient removal with mild scouring agents where it is desired to remove the conditioning agents or media after textile processing. On the other hand, if the compounds are permitted to remain upon the yarn after textile processing or fabrication procedures, their hydrophilic content is sufficiently manifest to permit effective and uniform dyeing of the textile materials by the customary dyeing procedures without interference therewith.

While these compounds have been found to exert a marked softening action upon textile materials of organic derivatives of cellulose, such softening action is not accompanied by any detrimental effect upon the tensile strength of the yarns or other textile material. For this reason, it is not thought likely that the compounds penetrate the organic derivative of cellulose mass in the manner of a plasticizer which tends to dissolve or be dissolved in the mass. Instead, the compounds are probably oriented or adsorbed on the surface of the masses, perhaps by residual valence forces. While their softening action is probably related to the hydrophilic portions of the molecules, yet the hydrophilic content of the compound does not appear to be sufficient to make them definitely hygroscopic. For this reason, the softening action is not appreciably affected by changes of humidity in the atmosphere and is independent of the method of application, that is, whether applied in the presence of aqueous or non-aqueous solvents whether volatile or non-volatile.

Examples of the compounds which are specified merely for the purpose of illustration are as follows:

Sorbitan or mannitan mono-, di-, and tri-oleates, monoricinoleates, stearates, palmitates, laurates, myristates, and sorbide or mannide mono-oleates, stearates, palmitates, laurates, myristates which may contain from zero to twenty or more mols of ethylene oxide condensed per molecule of ester.

These compounds, whether they contain or do not contain the ethylene oxide groups condensed therein, may be applied either from aqueous (except during dry-spinning) or non-aqueous media as they are readily emulsifiable in aqueous media and readily dispersible in non-aqueous media, such as mineral oil or volatile solvents. Such as these compounds as are liquids at ordinary room temperature may be applied directly without dilution or dispersion in other media, especially to freshly dry-spun filaments. Because of their solubility and dispersibility in such non-volatile liquid media as mineral oil, which itself can be applied in conjunction with the conditioning agent to increase the lubricity, all of these compounds may advantageously be applied in conjunction with such auxiliary agents, thereby eliminating the necessity to use expensive volatile solvents which would not keep the conditioning medium in permanent liquid condition and at the same time may tend to attack the textile materials of the organic derivatives of cellulose. As stated hereinbefore, this is also an important advantage in that it makes it possible to apply a substantially permanently blended liquid medium during dry spinning without

overstretching or difficulties with breakage. The application of such conditioning agents as a liquid or as a dispersion, solution, or emulsion readily lends itself to the uniform distribution of small amounts of the conditioning agents which may be as low as 0.1 to 5% based on the weight of the yarn, this low proportion being adequate for most purposes to soften and lubricate the yarns, fibers, or other textile materials, particularly freshly spun continuous filament yarns, and render them capable of being collected, amenable to subsequent processing operations or, instead, to impart the desired softness of hand to a finished fabric. Depending upon the conditions under which processing occurs, it may be desirable to add an antistatic agent, such as a higher fatty alcohol sulfate, to the conditioning agent or the solution, dispersion, or emulsion thereof to be applied. Other auxiliary agents may be incorporated as well, such as auxiliary emulsifying agents, or diluents to lower the viscosity. The conditioning agents may be applied in their liquid condition or in the form of solutions, dispersions, or emulsions by means of a wick, a coating roll, or by spraying or other customary methods.

Example 1

As an illustrative example, a freshly spun continuous filament yarn of cellulose acetate was passed in advance of its first collection over a coating roll dipping in a trough containing a solution in white mineral oil of 5% of mannitan mono-oleate containing three units of ethylene oxide condensed per molecule of the mannitan mono-oleate. The yarn thus treated was rendered soft, pliant and slippery and was collected without overstretching and breakage.

Example 2

Sorbitan mono-oleate condensed with 5 mols of ethylene oxide per mol of the ester was applied in liquid form to a freshly spun continuous filament yarn of cellulose acetate by means of an applicator roll in advance of its first collection preparatory to textile processing.

Example 3

A solution in white mineral oil of 7% sorbitan di-oleate containing 3 mols of ethylene oxide per molecule was applied to a running yarn of freshly spun continuous cellulose acetate filaments by a coating roll in advance of its first collection. The yarn thus conditioned was characterized by a soft hand, was pliant and slippery, and its textile processing qualities were of an excellent character.

Example 4

A solution in white mineral oil of 20% sorbitan di-oleate containing 8 mols of ethylene oxide per mol of the di-oleate and 2% of a higher fatty alcohol sulfate was applied by spraying to cellulose acetate staple fibers. The softened and lubricated fibers were then spun by the usual processes into a spun acetate yarn.

Example 5

A solution in white mineral oil of 5% of mannitan dilaurate and 2% of a higher fatty alcohol sulfate was applied while at spinning room temperature to a freshly dry-spun bundle of continuous cellulose acetate filaments as they proceeded from the spinning cell to a draw roll.

While preferred embodiments of the invention have been described, the description is intended to be illustrative only, and it is to be understood that changes and variations may be made without departing from the spirit or scope of the invention as defined by the appended claims.

I claim:

1. The process of conditioning freshly dry spun continuous filaments containing a cellulose organic ester which comprises applying thereto in advance of their first collection, at a room temperature between about 15° C. to about 45° C., a non-volatile, non-aqueous conditioning medium which is liquid throughout said range of room temperature, comprising a solution in a mineral oil vehicle of about 5 to 20% of an ethylene oxide condensation product of a partial higher fatty acid ester of an inner ether of a hexitol and a higher fatty alcohol sulfate in a relatively small proportion compared to the vehicle for imparting antistatic properties to the filaments, and thereafter collecting the filaments by winding.

2. The process of claim 1 in which the partial ester is derived from oleic acid.

3. The process of claim 2 in which the filaments are of cellulose acetate and the collection is accompanied by twisting.

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