

UNITED STATES PATENT OFFICE

2,068,003

TEXTILE PROCESS AND PRODUCT

George R. Blake and George W. Seymour, Cumberland, Md., assignors to Celanese Corporation of America, a corporation of Delaware

No Drawing. Application January 17, 1935,
Serial No. 2,205

18 Claims. (Cl. 28—1)

This invention relates to the treatment of yarn and other textile materials and relates more particularly to the conditioning of yarns made of organic derivatives of cellulose whereby the same may be successfully knitted into fabrics containing a large number of courses, or used in other textile operation in which the yarn is susceptible to a great number of very sharp bends as in circular knitting. An object of the invention is to incorporate in yarns containing organic derivatives of cellulose a conditioning fluid whereby the same are rendered more pliable and, therefore, more suitable for use in textile operations where they are subjected to rather sharp bends and turns. Other objects of the invention will appear from the following detailed description.

The textile operations which require the yarn to be flexed several times and through very sharp angles as found in circular and warp knitting, present serious difficulties such as stitch distortion due to the lack of pliability of the yarn or the fluffing of the yarn passing through the needles and guides of the machine. In order to condition such yarns animal, vegetable oil such as olive oil, or mineral oil have been applied thereto. Further, compounds containing animal, vegetable or mineral oils compounded with glycerides, fatty acids and other materials have been applied to yarn. While these materials render such yarns pliable to a certain degree, after storage of the yarn for a period of time they develop either a gumminess on the yarn or a harshness in the yarn. Also yarns lubricated with these materials in the customary manner and to the customary degree are difficult to circular knit into a fabric containing 45 or more courses per inch.

We have found that if there is incorporated in the yarn a sufficient quantity of conditioning fluid containing a softening agent for the organic derivative of cellulose and a lubricant, the yarns containing organic derivatives of cellulose may successfully be fabricated by circular knitting or otherwise into fabrics of very close stitch shape and that during such fabrication there is neither stitch distortion nor a breaking into holes due to the yarn breaking where they are bent through a very sharp angle. Moreover, yarns treated with such a lubricant and to such a degree may be stored for periods of a few weeks before fabricating them to a fabric without materially affecting the resulting product.

In accordance with our invention, we incorpo-

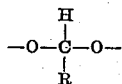
rate in or apply as a coating to textile materials comprising filaments of cellulose acetate or other organic derivatives of cellulose at least 15%, based on the weight of the textile material, of a conditioning agent containing an ether of the type hereinafter defined, which is a solvent or at least a latent solvent for the cellulose acetate or other organic derivatives of cellulose, compounded with a lubricant material containing vegetable oils, animal oils and/or oxidized oils.

The textile materials to be made or treated in accordance with our invention may be in the form of yarns containing a plurality of filaments made of cellulose acetate or other organic derivative of cellulose. These yarns may be composed of any number of individual filaments or the same may be composed of short lengths of filaments, for instance, staple fibers twisted together to form a yarn. Any degree of twist may be employed in the yarns. While we prefer to treat textile materials containing cellulose acetate, such textile materials may contain other organic derivatives of cellulose such as cellulose formate, cellulose propionate, cellulose butyrate, ethyl cellulose, methyl cellulose and benzyl cellulose. If desired, the yarns or filaments thereof made or treated in accordance with this invention may have finely divided pigment like material such as titanium dioxide, antimony trioxide, stannic oxide, starch, high melting point insoluble organic compounds and the like incorporated therein in order to give them a subdued luster.

Yarns to be treated need not consist wholly of filaments of cellulose acetate but may also contain fibers of other materials such as natural silk, artificial fibers of reconstituted cellulose, wool, cotton, etc. Yarns of cellulose acetate may be formed from continuous filaments or the same may be spun from short staples or short lengths of such filaments.

The principal object of the present invention is to condition yarns comprising organic derivatives of cellulose, i. e. to treat such yarns with a suitable fluid so as to render them more pliable, sufficiently tacked together and sufficiently lubricated and, therefore, more suited for use in textile operations requiring very fine stitch shapes or sharp bends of the material. It also results in an even tension on the yarns as they pass through needles, guides, tensioning devices, etc. of the fabricating machines. In accordance with the invention, such conditioning is effected by treating the yarns with a compound which may be termed a lubricating compound. This

compound is preferably made of two or more ingredients, one of which has a softening action or solvent action upon the organic derivative of cellulose and the other may or may not have these properties but has the property of lubricating the yarn and wetting same. The solvent or softening agent to be incorporated in the lubricating compound is preferably a type of ether. The type of ether referred to is an ether of the formula



where R is hydrogen or an alkyl or phenyl residue and the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid, and obtainable by the condensation of an aldehyde such as formaldehyde, acetaldehyde, butyraldehyde or benzaldehyde with an aliphatic hydroxy compound, for example, a polyhydric alcohol, a partial ether of a polyhydric alcohol, or an aliphatic hydroxy carboxylic acid. This type of ether is referred to hereinafter in the specification and in the claims as a formal-ether.

The formal-ether of the present invention may be made by the condensation of formaldehyde, formalin, tri-hydroxy-methylene, butraldehyde or paraformaldehyde in the presence or absence of an acid catalyst with simple polyhydric alcohols, such as glycerol, ethylene glycol or propylene glycol, or substituted polyhydric alcohols such as glycerolmono-chlorohydrine or di-ethylene glycol. Examples of partial ethers of polyhydric alcohols that may be employed for making the formal-ether of the type above defined are the mono-methyl ether of ethylene glycol, the mono-ethyl ether of ethylene glycol, and the mono-methyl or ethyl ether of propylene glycol or of diethylene glycol. Examples of hydroxy carboxylic acids that may be used for preparing the formal-ethers of the type above defined are alpha-hydroxy-iso-butyric acid, alpha-hydroxy normal butyric acid, glycollic acid, lactic acid, etc. Examples of acid catalysts that may be employed for promoting the condensation are ferric chloride or other acid salts, hydrochloric acid, sulphuric acid, phosphoric acid or other strong or weak mineral acids.

Generally, it is preferable to employ these formal-ethers of the type hereinbefore defined that are relatively non-volatile, having a boiling point above, say, 140° C. and are soluble in or miscible with water. The reason that relatively non-volatile formal-ethers are preferred is that upon the fabricating of the conditioned yarn such formal-ethers do not evaporate readily and, therefore, the treated yarns retain their pliability for a long period of time. Since it is desirable that the formal-ethers which are solvents or latent solvents be removed from the fabric after it is formed from the treated yarn, if they are water soluble they can be readily removed in the ordinary scouring and/or dyeing operations and even exert a beneficial effect in such operations.

If desirable, the formal-ethers of the type hereinbefore defined may be employed in conjunction with other relatively high boiling solvents or latent solvents for organic derivatives of cellulose, such as ethyl oxybutyrate, benzyl alcohol, di-acetone alcohol, monacetin, diacetin,

acetyl carbinol, cyclopentanone, di-methyl tartrate, etc.

While one or more formal-ethers of the type hereinbefore defined may be incorporated in or applied to the textile materials alone, generally it is preferable to apply the same in admixture with one or more other conditioning agents, such as oil lubricants and relatively non-volatile materials having an affinity for water. Since it is desirable that the organic derivative of cellulose yarn have thereon an oily lubricant to facilitate winding, twisting, knitting, weaving or other textile operations, it is preferred to incorporate in the conditioning or lubricating compounds along with the formal-ethers an oily material. The oily material may be any suitable oil or materials having the property of lubrication, it being preferred, however, to use a high percentage of an oil which does not readily oxidize or otherwise change its properties to form large quantities of free acid on one hand or to polymerize and form gums on the other hand. Examples of suitable oils are olive oil, castor oil, teaseed oil, egg oil, soy bean oil, peanut oil, palm oil, cottonseed or other non-drying oils or other vegetable, animal or mineral oils. If the specific oils employed are not miscible with the specific formal-ether used they may generally be rendered miscible with each other and with any polyhydric alcohol and/or water present by the addition of a free fatty acid such as oleic acid or stearic acid, or a cyclic hydrocarbon such as xylol.

The tendency of the oily material in the lubricating compound to oxidize or otherwise change its properties is retarded by the presence of a formal-ether. It is of advantage, however, to employ pre-oxidized vegetable oils, such as oxidized olive oil, oxidized castor oil, etc. The difficulty of ageing of the ordinary oils is thus largely overcome and, moreover, such oxidized oils have greater miscibility with the formal-ether or a mixture of the formal-ether and a polyhydric alcohol in water than do normal oils. The oxidized oil may be prepared in any suitable manner, for instance, by injecting a fine stream of oxygen into a body of olive oil or more preferably by injecting olive oil in a fine spray into a stream of oxygen until the required degree of oxidation has occurred.

As to the relative proportions of formal-ether to olive or other oil employed, these may vary within relatively wide limits depending upon the results desired, the proportions being generally from 50 to 90% of formal-ether and 50 to 10% of olive oil or other oil.

To the yarn, as a separate conditioning liquid or as a liquid mixed with the compounded lubricant, there may be applied a hygroscopic relatively non-volatile substance. This hygroscopic substance may be applied subsequently to the lubricant either with or without formal-ether. Examples of such hygroscopic substances are relatively non-volatile liquids having an affinity for water, such as di-ethylene glycol, glycerine, ethylene glycol, propylene glycol or other polyhydric alcohols, the mono-ethyl ether of ethylene glycol and other ethers of glycols or other polyhydric alcohols. Generally, these will be applied in association with water, the amount of water present preferably being such that it forms a solution which, upon exposure to air, does not change appreciably in concentration. The amount of water may be from 20 to 50% the weight of the hygroscopic liquid.

The conditioning fluid or the compounded lu-

bricant either with or without the hygroscopic material may be incorporated in the textile material in any suitable manner. Thus it may be added to the spinning solution containing the cellulose acetate dissolved in a volatile solvent in amounts from 0.5% to 15% of the weight of the cellulose acetate present and the spinning solution may then be extruded through orifices into an evaporative atmosphere.

As to the amount of conditioning fluid containing the formal-ether that may be applied to the yarn, this is very important when desiring to circular knit fabrics containing a large number of courses per inch. For example, in knitting the fabric that is to contain above 40 courses per inch in the finished fabric at least 15% of the conditioning agent calculated as a mixture of formal-ether and vegetable oil is required. For best results in forming the fine stitch shapes from 15 to 25% or more of the conditioning liquid is required. Circular knit fabrics may be made having as high as 56 to 60 courses per inch when employing yarns containing 20% of the conditioning liquid consisting of formal-ether, oxidized olive oil and olive oil. It is found that the yarn must be wet to the point of saturation with the conditioning fluid for the best results.

The conditioning fluid may be applied to the yarn in any suitable manner. Thus it may be applied by the immersing of hanks of yarn to be treated in a bath containing the conditioning fluid. If desired, the conditioning fluid may be applied to the yarn while it is in transit from package to package. This may be done by passing the yarn over pads or wicks that are impregnated with the conditioning fluid or by passing over rollers or discs, the bottoms of which are immersed in the conditioning fluid. The conditioning fluid may be dripped onto the yarns while they are in transit, and also the yarns may be caused to dip into a bath containing the conditioning fluid while they are in transit, prior to being wound onto a suitable package. In another form, the conditioning fluid may be applied as a spray onto cones, bobbins, pirns or other packages while the same are being wound, so that the spray is being continuously applied during the formation of the package. If desired, the yarn may be treated at the spinning cabinet with the conditioning agent while it is being transmitted from the spinning cabinet in which it is spun from solutions of organic derivatives of cellulose and prior to being wound and/or wound and twisted into yarn.

The yarn treated in accordance with this invention is very pliable and flexible, and may be knitted, woven or subjected to any other suitable textile operations, to form knitted or woven fabric, hosiery, or other articles.

Cellulose acetate yarns treated in accordance with this invention have many important advantages. As stated, they are very pliable and flexible, and even after storage for a prolonged period of time, not only is this pliability or flexibility retained but in many instances is actually improved. Therefore irrespective of the age of the treated yarns, they can be readily rewound from their original package or subjected to any textile operation.

This yarn may be knitted on circular knitting machines or on warp knitting machines to produce fabrics of many wales and courses per unit length, which fabrics are substantially free of pin holes, distortions, tension lines, rowiness, etc., and have a much better appearance than fabric

made from untreated yarns. Cellulose acetate yarn when treated by this invention may be knitted on certain circular knitting machines which are not capable of knitting untreated cellulose acetate yarn or cellulose acetate yarn finished by previous methods, to form a commercially satisfactory fabric and likewise may be netted successfully on lace-making machines to form highly satisfactory lace. Moreover, when yarn treated by this invention is exposed for several days to normal atmospheric conditions without any precautions whatever, the yarn still retains its good knitting properties. Furthermore, yarn treated by this invention may be knitted at a higher rate than yarn treated by previous methods. The yarn may be processed regardless of humidity conditions and this renders the use of expensive humidifying apparatus unnecessary.

In order further to illustrate our invention and without being limited thereto, the following specific example is given:

Example

Conditioning liquid is made as follows:—

Parts by weight	
The ether formed by the condensation of formaldehyde with the monomethyl ether of ethylene glycol.....	75 to 85
Oxidized olive oil.....	12 to 20
Olive oil.....	3 to 8

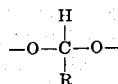
This conditioning liquid may be applied by means of wicks, rollers, discs or other suitable furnishing devices to the cellulose acetate filaments immediately after their leaving the dry spinning machine and prior to their being wound or twisted and wound, or it may be applied to the yarns during any subsequent textile operation. Yarn intended for fine knitting purposes may be conditioned with the above liquid by applying thereto 18 to 20% of the liquid based on the weight of the organic derivative of cellulose in the yarns. As this amount of lubricating compound forms a considerably thick coating, it is preferable to knit the yarn within 25 days after the application of the lubricating compound thereto or to apply an additional lubricant just before knitting.

Yarns of organic derivatives of cellulose may be formed containing therein from 3 to 10% of the conditioning liquid and then treated before knitting with from 10 to 20% as a coating to condition the yarns for perfect knitting purposes.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit of our invention.

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

1. Yarn containing an organic derivative of cellulose and more than 15% on the weight of the organic derivative of cellulose of a lubricating compound comprising an ether having the formula



where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from

polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oil.

2. Yarn containing cellulose acetate and more than 15% on the weight of the cellulose acetate of a lubricating compound comprising an ether having the formula



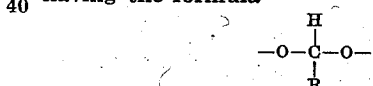
where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oil.

3. Yarn containing an organic derivative of cellulose and more than 15% on the weight of the organic derivative of cellulose of a lubricating compound comprising an ether having the formula



where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oxidized oil.

4. Yarn containing cellulose acetate and more than 15% on the weight of the cellulose acetate of a lubricating compound comprising an ether having the formula



where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oxidized oil.

5. Yarn containing an organic derivative of cellulose and more than 15% on the weight of the organic derivative of cellulose of a lubricating compound comprising at least 50% of an ether having the formula



where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oil.

6. Yarn containing cellulose acetate and more than 15% on the weight of the cellulose acetate of a lubricating compound comprising at least 50% of an ether having the formula

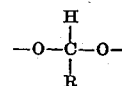


where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oil.

7. Yarn containing an organic derivative of cellulose and more than 15% on the weight of the organic derivative of cellulose of a lubricating compound comprising the condensation product of formaldehyde with the mono-methyl ether of ethylene glycol, and an oil.

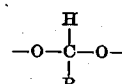
8. Yarn containing cellulose acetate and more than 15% on the weight of the cellulose acetate of a lubricating compound comprising the condensation product of formaldehyde with the mono-methyl ether of ethylene glycol, and an oil.

9. Yarn containing an organic derivative of cellulose and from 18 to 20% on the weight of the organic derivative of cellulose of a lubricating compound comprising an ether having the formula



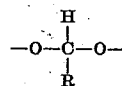
where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oil.

10. Yarn containing cellulose acetate and from 18 to 20% on the weight of the cellulose acetate of a lubricating compound comprising an ether having the formula



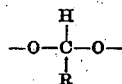
where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oil.

11. A fabric formed from yarns containing organic derivatives of cellulose and at least 15% of a lubricating compound containing an ether having the formula



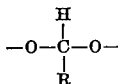
where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oil.

12. A fabric formed from yarns containing cellulose acetate and at least 15% of a lubricating compound containing an ether having the formula



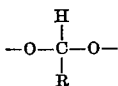
where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oil.

13. Yarn containing organic derivative of cellulose filaments containing a metallic salt, said yarn having a coating of at least 15% its weight of a lubricating compound containing an ether having the formula



where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oil.

14. Yarn containing cellulose acetate filaments containing a metallic salt, said yarn having a coating of at least 15% its weight of a lubricating compound containing an ether having the formula



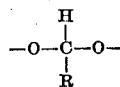
where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid and an oil.

15. Yarn containing an organic derivative of cellulose and more than 15%, based on the weight of the derivative of cellulose present, of a lubricating compound comprising an oil, an oxidized oil and an ether having the formula

where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid.



16. Yarn containing cellulose acetate and more than 15%, based on the weight of the cellulose acetate present, of a lubricating compound comprising an oil, an oxidized oil and an ether having the formula



where R is hydrogen or an alkyl or phenyl residue, and where the two free oxygen bonds are linked to two monovalent radicals derived from polyhydric alcohols or from hydroxy-carboxylic acids or are linked to a divalent radical derived from a polyhydric alcohol or a hydroxy-carboxylic acid.

17. Yarn containing an organic derivative of cellulose and more than 15%, based on the weight of the organic derivative of cellulose present, of a lubricating compound comprising the condensation product of formaldehyde with the mono-methyl ether of ethylene glycol, olive oil and oxidized olive oil.

18. Yarn containing cellulose acetate and more than 15%, based on the weight of the cellulose acetate present, of a lubricating compound comprising the condensation product of formaldehyde with the mono-methyl ether of ethylene glycol, olive oil and oxidized olive oil.

GEORGE R. BLAKE.

GEORGE W. SEYMOUR.