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TEXTILE PROCESS AND PRODUCT

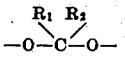
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29 Claims. (Cl. 28—1)

This invention relates to the treatment of yarn and other textile materials and relates more particularly to the conditioning of yarn made of organic derivatives of cellulose whereby the same may be more successfully subjected to knitting or other textile operations.

The principal object of the present invention is to condition yarns comprising organic derivatives of cellulose, i. e., to treat said yarns with a suitable fluid so as to render them more pliable and therefore more suited for use in textile operations, such as knitting, where they are subjected to rather sharp bends and turns. According to the invention such conditioning is effected by treating the yarns with a particular type of ether. The type of ether referred to is an ether containing the grouping



and obtainable by the condensation of an aldehyde such as formaldehyde, acetaldehyde 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 claims as a 1.3-diether. Other objects of this invention will appear from the following detailed description.

The knitting of yarns made of organic derivatives of cellulose presents serious difficulties, since if attempts are made to form a closely knit fabric from such yarns, on certain machines, such as a Wildman circular knitting machine forming a straight stitch, serious defects or faults develop because of the lack of pliability of such yarns. In order to condition such yarns, animal or vegetable oil such as olive oil, or mineral oils have been applied thereto. While olive oil renders such yarn pliable to a certain degree, after a storage of the yarn for a period of time, yarn coated therewith does not knit as well as is often desired. Moreover the frictional resistance of the yarn treated with olive oil increases very rapidly. Mineral oils do not impart pliability to as great an extent as may be desired, and moreover are not readily removed from the fabric made from such yarns by the ordinary scouring baths.

We have found that if there is incorporated in yarn containing cellulose acetate or other organic derivatives of cellulose a conditioning fluid containing a formal that is a solvent or at least a latent solvent for the cellulose acetate or other organic derivative of cellulose, the resulting yarn has highly increased flexibility, and knit fabrics of close construction may be formed therefrom, which fabrics are substantially free from defects occurring in fabrics made from insufficiently conditioned yarn. Moreover upon storage of such treated yarn, the knitting and other properties of the same are not deleteriously affected, and in many cases are even improved after a period of time. Other advantageous properties are also imparted to the yarn as will appear below.

In accordance with our invention we incorporate in textile materials comprising filaments of cellulose acetate or other organic derivatives of cellulose, a conditioning agent containing an ether of the type hereinbefore defined that is a solvent or at least has a latent solvent action on the cellulose acetate or other organic derivative of cellulose.

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 derivatives of cellulose. On the other hand the textile materials may be in the form of films, artificial bristles or straw containing such cellulose acetate so as to make it possible to knot, bend or braid them more easily. 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 other textile material made or treated in accordance with this invention may have finely divided pigment-like material, such as titanium dioxide, antimony trioxide, stannic oxide, starch, highly melting insoluble organic compounds and the like, incorporated therein, whereby they have a subdued luster.

The yarn to be treated need not consist wholly of yarns of cellulose acetate but may also contain fibres of other materials such as natural silk, artificial fibres of reconstituted cellulose, wool, cotton, etc. The 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 ethers of the present invention may be made by the condensation of formaldehyde, formalin, trihydroxy-methylene or para formaldehyde in the presence or absence of an acidic catalyst with simple polyhydric alcohols, such as glycerol, ethylene glycol or propylene glycol, or substituted polyhydric alcohols such as glycerol mono chlorhydrin or diethylene glycol. Examples

of partial ethers of polyhydric alcohols that may be employed for making the ethers 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 ethers of the type above defined are α-hydroxy-iso-butyric acid, α-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, sulfuric acid, phosphoric acid or other strong or weak mineral acids.

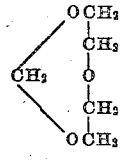
Representative examples of methods of making some of these ethers will be given merely by way of illustration, it being understood that this invention is of general application and is in no way limited to the specific mode of preparing the ethers employed therein.

The following is a method of preparing one of the ethers of the present invention.

Two molecular proportions of the monomethyl ether of ethylene glycol, $CH_3OCH_2CH_2OH$, one (1) molecular proportion of para-formaldehyde and hydrated ferric chloride (in amount equal to 4% of the weight of the reactants) are subjected to slow distillation. The distillate is collected in three fractions, namely at temperatures from 92 to 110° C., from 110 to 180° C. and from 180 to 200° C. Calcium chloride is added to the first two fractions and the upper layers thereof are added to the third fraction and the mixture is then redistilled. The fraction boiling at 197° to 205° C. is collected, and this is an ether which is probably represented by the formula

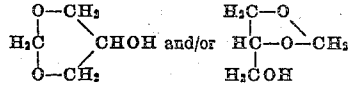
$$(CH_3OCH_2CH_2O)_2CH_2$$

Another ether of the present invention, the methylene ether of diethylene glycol, having the probable structural formula of



may be formed by refluxing for 18 hours, 106 grams of diethylene glycol with 30 grams of paraformaldehyde in the presence of 5 cc. of concentrated HCl. The resulting material is fractionally distilled and the fraction boiling between 180 to 240° C. is separated.

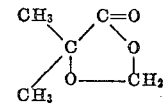
Another ether of the type hereinbefore defined which has the structural formulae of



may be prepared, for instance, by refluxing equimolecular proportions of glycerol or polyglycerol with formalin, tri-hydroxymethylene or paraformaldehyde in the presence of hydrochloric acid (HCl) or other suitable catalyst.

The following method of preparing the diether of the α-hydroxy-iso-butyric acid is illustrative of the method of preparing the diethers of hydroxy carboxylic acids.

One (1) molecular proportion of α-hydroxy-iso-butyric acid is mixed with one (1) molecular proportion of paraformaldehyde and a very small amount of sulfuric acid as catalyst is added thereto. The mixture is subjected to slow distillation. The major portion of the distillate comes over at about 110° C. and separates into two layers. The lower layer is dried and redistilled, the distillate being a product boiling at 148 to 149° C. and having the probable formula



Generally we prefer to employ those ethers of the type hereinbefore defined that are relatively non volatile having a boiling point above say 140° C. and that are soluble in or miscible with water. The reason that relatively non-volatile diethers are preferred is that upon storage of the conditioned yarn such diethers do not tend to evaporate readily and therefore the treated yarns retain their pliability for a long period of time. Since it is desirable that the diethers 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 desired the ethers of the type hereinbefore defined may be employed in conjunction with other relatively high boiling solvents or latent solvents for cellulose acetate, such as ethyl oxy butyrate, benzyl alcohol, diacetone alcohol, monacetin, diacetin, acetol (acetyl carbinol) cyclohexanone, cyclopentanone, dimethyl tartrate, etc.

While one or more ethers of the type hereinbefore defined alone may be incorporated in or applied to the textile materials, generally it is preferable to apply the same in admixture with one or more other conditioning agents such as oily 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 will be found advantageous to incorporate such oils in the conditioning liquid containing the ethers of the type hereinbefore defined, unless the oily lubricant has already previously been applied to the textile material that is to be treated with the conditioning liquid. Thus olive oil, castor oil, soya bean oil, peanut oil, palm oil, cotton seed or other non-drying oils or other vegetable, animal or mineral oils may be admixed with the ether of the present invention when applied to the textile material. If the specific oils employed are not miscible with the specific ether of the present invention 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.

While olive oil, when applied to yarn, tends to oxidize or harden with time, thus rendering difficult the rewinding, knitting or other manipulation of aged yarn, the presence of the ether of the present invention overcomes this difficulty largely or entirely since the ether of the present invention forms a solution with such oxidized products.

We have further found that if pre-oxidized vegetable oils such as olive oil, castor oil or other so called non-drying oils are employed as lubricants, the difficulty of ageing of the ordinary oils is largely overcome and moreover such oxidized oils have greater miscibility with the ether of the present invention, or a mixture of ether of the present invention, a polyhydric alcohol and water

than do the normal oils. The oxidized oil may be prepared in any suitable manner, e. g. 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 ethers of the present invention 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 80% of ethers of the present invention and 50 to 20% of olive or other oil. If the yarn is to be employed for knitting or like purposes the higher proportions of ethers of the present invention will be employed, while if the yarn is to be employed for weaving or similar purposes, the lower proportions of ethers of the present invention will be used.

If the yarn is to be knitted or subjected to similar severe textile operations, we prefer to apply thereto a hygroscopic relatively non volatile substance, which may be incorporated in the conditioning liquid containing the ethers of the present invention or if such ethers of the present invention have previously been applied to the yarn, this hygroscopic substance may be applied subsequently either with or without the ethers of the present invention. Examples of such hygroscopic substances are relatively non-volatile liquids having an affinity for water such as diethylene glycol, glycerin, ethylene-glycol, propylene glycol or other polyhydric alcohols, the mono ethyl ether of ethylene glycol and others 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 forms a solution which upon exposure to air does not change appreciably in concentration. The amount of water may be from 20 to 50% of the hygroscopic liquid present.

The ethers of the type hereinbefore defined may be incorporated in the textile material in any suitable manner. Thus they 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 ether of the present invention that may be applied to the yarn, this will vary with the purposes to be attained and the composition of the conditioning fluid. If the yarn treated is to be subjected to a knitting or analogous textile operation, the amount of conditioning liquid applied may be from 3 to 15% of the weight of the yarn, the larger percentage being applied in those cases where the conditioning fluid contains smaller percentages of the ether of the present invention. If the conditioning fluid is to be applied to the yarn which is to be woven, the amount of conditioning fluid applied may be only 1 to 3%.

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.

After the fabric is formed, it is highly desirable that the ethers of the present invention be completely removed from the fabric by scouring or during dyeing or any other finishing operation. We have found that the ethers of the present invention which are solvents or latent solvents tend to lower the melting point or safe ironing point of the material when they are present, but after they are removed by any suitable means, the resulting textile material has as high a melting point and safe ironing point as the untreated textile material.

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.

Yarns treated by this invention or fabrics made therefrom dye more readily since the ether present permits the rapid wetting out and penetration of the fibres due to their softened nature. Moreover fabrics made of such yarn do not tend to develop wrinkles or creases when dyed, de-

lustered, scoured or subjected to other finishing treatments, a factor which is of great importance in the case of hosiery or other knitted wares and in the case of dyeing or delustering of woven fabrics on a winch or similar apparatus where the material is treated in folded form. Yarns or fabrics treated with or containing the ethers of the present invention deluster more readily and evenly when subjected to hot aqueous media such as soap solutions at or near the boiling point of water.

Yarns treated in accordance with this invention produce woven fabrics that are substantially free of warp streaks or weft bars in contrast with yarn lubricated by prior methods. Fabrics produced from such yarn have less tendency to initial shrinkage thus giving greater flexibility in the final finished width without encountering subsequent shrinkage.

In order further to illustrate our invention but without being limited thereto, the following specific examples are given.

Example I

A conditioning liquid is made as follows:

	Parts by weight
The ether formed by the condensation of formaldehyde with the mono methyl ether of ethylene glycol	60 to 70
Oxidized olive oil	40 to 30
A mixture of 70% diethylene glycol and 30% of water	6 to 12

This conditioning liquid may be applied by means of wicks, rollers, discs or other suitable furnishing devices to the cellulose acetate filaments immediately after 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. If the yarn is intended primarily for knitting purposes the amount of conditioning liquid applied will be from 4 to 6% of the weight of the yarn. If the yarn is intended primarily for weaving the amount of conditioning liquid applied may be only 1 to 2% of the weight of the yarn and the conditioning liquid will contain a smaller proportion of the diether.

Example II

A conditioning liquid is made up as follows:

	Parts by weight
The ether formed by the condensation of formaldehyde with the mono methyl ether of ethylene glycol	70
Ordinary olive oil	30

This conditioning liquid may be applied to the cellulose acetate filaments immediately after their leaving the dry spinning cell in which they are formed and prior to their being twisted and wound. If the yarn is to be used for weaving the amount of conditioning liquid applied may be 1 to 2% of the weight of the yarn.

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 claim and desire to secure by Letters Patent is:

1. Method of conditioning textile materials containing organic derivatives of cellulose comprising incorporating a conditioning agent comprising a 1.3 diether therein.

2. Method of conditioning textile materials containing cellulose acetate comprising incorporating a conditioning agent comprising a 1.3 diether therein.

3. Method of conditioning yarn containing organic derivatives of cellulose comprising applying thereto a conditioning agent comprising a relatively non-volatile 1.3 diether that has a softening action on the organic derivative of cellulose.

4. Method of conditioning yarn containing cellulose acetate comprising applying thereto a conditioning agent comprising a relatively non-volatile 1.3 diether that has a softening action on the cellulose acetate.

5. Method of conditioning yarn containing organic derivatives of cellulose comprising applying thereto a conditioning agent comprising a relatively non volatile 1.3 diether of a compound selected from the group consisting of polyhydric alcohols and their partial ethers.

6. Method of conditioning yarn containing cellulose acetate comprising applying thereto a conditioning agent comprising a relatively non-volatile 1.3 diether of a compound selected from the group consisting of polyhydric alcohols and their partial ethers.

7. Method of conditioning yarn containing cellulose acetate comprising applying thereto a conditioning agent comprising the 1.3 diether of the formula $(CH_3OCH_2CH_2O)_2CH_2$.

8. Method of conditioning yarn containing organic derivatives of cellulose comprising applying thereto a conditioning agent comprising a 1.3 diether and an oil.

9. Method of conditioning yarn containing cellulose acetate comprising applying thereto a conditioning agent comprising a relatively non volatile 1.3 diether and an oxidized non drying oil.

10. Method of conditioning yarn containing cellulose acetate comprising applying thereto a conditioning agent comprising a relatively non volatile 1.3 diether and oxidized olive oil.

11. Method of conditioning yarn containing cellulose acetate comprising applying thereto a conditioning agent comprising 50 to 80 parts by weight of a relatively non volatile 1.3 diether and 50 to 20 parts by weight of an oil.

12. Method of conditioning yarn containing cellulose acetate comprising applying thereto a conditioning agent comprising a relatively non volatile 1.3 diether, an oil and a hygroscopic substance.

13. Method of conditioning yarn containing cellulose acetate comprising applying thereto a conditioning agent comprising the 1.3-diether of the formula $(CH_3OCH_2CH_2O)_2CH_2$, oxidized olive oil, diethylene glycol and water.

14. Textile materials comprising organic derivatives of cellulose having therein a conditioning agent comprising a 1.3 diether.

15. Textile materials comprising cellulose acetate having therein a conditioning agent comprising a relatively non volatile 1.3 diether.

16. Yarn comprising organic derivatives of cellulose having applied thereto a conditioning agent comprising a relatively non volatile 1.3 diether and an oil.

17. Yarn comprising cellulose acetate having applied thereto a conditioning agent comprising a relatively non-volatile 1.3 diether, oxidized olive oil and diethylene glycol.

18. Method of conditioning yarn containing organic derivatives of cellulose which comprises incorporating therein a conditioning agent comprising a cyclic 1.3 diether.

19. Method of conditioning yarn containing cellulose acetate which comprises incorporating therein a conditioning agent comprising a cyclic 1.3 diether.

20. Method of conditioning yarn containing organic derivatives of cellulose which comprises applying thereto a conditioning agent comprising a cyclic 1.3 diether and an oil.

21. Textile materials containing organic derivatives of cellulose having therein a conditioning agent comprising a cyclic 1.3 diether.

22. Textile materials containing cellulose acetate having therein a conditioning agent comprising a cyclic 1.3 diether.

23. Textile material comprising organic derivatives of cellulose having therein an ether formed by the condensation of formaldehyde with the mono methyl ether of ethylene glycol.

24. Textile material comprising cellulose acetate having therein an ether formed by the condensation of formaldehyde with the mono methyl ether of ethylene glycol.

25. Textile material comprising cellulose acetate having therein an ether formed by the condensation of formaldehyde with the mono methyl ether of ethylene glycol, and an oxidized olive oil.

26. Textile material comprising organic derivatives of cellulose having a 1.3-diether and oxidized olive oil therein.

27. Textile material comprising cellulose acetate having a 1.3-diether and oxidized olive oil therein.

28. Textile materials comprising organic derivatives of cellulose having a 1.3-diether and oxidized non-drying oils therein.

29. Textile materials comprising cellulose acetate having a 1.3-diether and oxidized non-drying oils therein.

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