

# UNITED STATES PATENT OFFICE

2,418,752

## YARN HAVING THE TWIST SET THEREIN WITH AN UNCTUOUS SOLID

Kenneth R. Brown, West Chester, Pa., assignor,  
by mesne assignments, to American Viscose  
Corporation, a corporation of Delaware

No Drawing. Application April 24, 1943,  
Serial No. 484,493

16 Claims. (Cl. 57—140)

1

This invention concerns the conditioning or preparation of textile materials such as filaments, fibers, yarns and the like, for textile operations, such as spinning, throwing, twisting, knitting, weaving, braiding and the like.

It is an object of the invention to provide filaments, fibers, yarns and the like (hereinafter referred to as "filamentary material") with an improved conditioning material which is easy to apply and is readily removed from the filamentary material when it is no longer needed, which imparts pliability, softness and lubricity to the treated filamentary material, and which is capable of setting the twist and maintaining its uniformity, particularly in highly twisted yarns, and at the same time is free of any tendency to be stripped from the filamentary material while it is being subjected to the severest flexing and rubbing action during the course of textile processing, such as in knitting or spinning.

It has been found that these results can be satisfactorily fulfilled by the application, to the yarns and the like, of unctuous to waxy solids having melting points ranging between 30° C. and about 70° C. or higher comprising higher fatty acid esters of ethers of polyols having at least three hydroxyl groups formed by condensation of such a polyol with an olefine oxide preferably having no more than 3 carbon atoms. The term "polyol" is meant to include only polyhydric alcohols and carbohydrates. These compounds may be prepared, by way of illustration, by first condensing in the presence or absence of water an olefine oxide, such as ethylene oxide or propylene oxide, with one or more of such polyhydric alcohols as glycerol, diglycerol, pentaerythritol, erythritol, sorbitol, sorbitan, mannitol, inannitan, dulcitol, dulcitan or of related polyols including the carbohydrates, such as glucose and other sugars, and then subjecting the condensation product to esterification with a higher saturated fatty acid having at least 16 carbon atoms in a straight chain or a mixture of one or more of such higher fatty acids with a relatively small proportion of one or more higher unsaturated fatty acids. The maximum proportion of unsaturated fatty acid that may be used depends upon the particular acid, the nature and amount of saturated fatty acid used and the nature and amount of alkylene oxide combined in the molecule. Generally at least two thirds or more of the acyl radicals in the final compound must be saturated. The esterification contemplated by the invention ranges from about 70% of the calculated free hydroxyl groups in the ether up to

2

complete esterification thereof. The fatty acids may be those obtained by the saponification of palmitin, stearin and the like or of animal and vegetable oils, such as fish oils, olive oils, lards, or fractions thereof having the desired long carbon chain and the subsequent hydrogenation of such of the above-mentioned fatty acids as are highly unsaturated to complete saturation or at least to attain the ratio specified above between the saturated and unsaturated acids. The esters of the condensation products obtained from substantially pure compounds as well as those obtained from commercial grades of the several compounds have both been found to function satisfactorily. In addition, a mixture of two or more of the pure polyhydric alcohols may be reacted with a mixture of two or more olefine oxides.

The proportion of alkylene oxide that is present in the compounds may be varied considerably provided there are not so many olefine oxide units per mol of the compound as to make it of too low a melting point. The permissible proportion of olefine oxide depends upon the particular oxide as well as the molar proportion of the ester radicals present therein and the particular ester radical and especially upon the ratio between saturated and unsaturated acids. For example, sorbitol can tolerate up to about 20 mols of oxyethylene units per mol if the product is then completely esterified with stearic acid, whereas only about one third this number of oxypropylene groups is permissible. Where a corresponding palmitic or oleic ester compound is involved, the upper limit of oxyolefine groups is correspondingly reduced.

The compounds contemplated by this invention are unctuous and waxy solids having melting points between 30° C. to about 70° C. Depending upon the ambient temperature where the treated yarns are to be subjected to textile operations, a compound having a higher or a lower melting point (or titer) is to be preferred. For a given ambient temperature, a compound should be selected which remains solid at that temperature and which is only superficially melted, if at all, by the friction involved in passing through the guides, knitting needles, and the like. At the same time, the compound, should not have such a high melting point that excessively high temperatures, which might harm the textile materials, are required in the scouring bath to effect melting and thereby hasten emulsification. Those compounds having lower melting points in the neighborhood of about 35° C. to 45° C. render exceptional satisfaction under normal

conditions of service. The following table sets forth illustrative examples, giving the number of mols of olefine oxide condensed with one mol of the polyhydric alcohol and the number of mols of acid or mixed acids with which the condensation product is esterified:

No.	Polyhydric Alcohol	Mols of Olefine Oxide	Mols of Fatty Acid
1	sorbitol	4 ethylene oxide	6 stearic.
2	do	6 ethylene oxide	Do.
3	sorbitol (commercial grade)	do	Do.
4	sorbitol	do	6 mixed acids, 75% stearic, balance chiefly palmitic.
5	do	do	6 hydrogenated fatty acids derived from fish oil.
6	do	do	6 hydrogenated fatty acids from soya bean oil.
7	do	do	6 hydrogenated fatty acids from cottonseed oil.
8	do	do	6 palmitic.
9	do	do	1 oleic mixed with 5 hydrogenated fatty acids from soya bean oil.
10	do	do	1 lauric mixed with 5 hydrogenated fatty acids from cottonseed oil.
11	do	do	1 abetic mixed with 5 hydrogenated fatty acids from fish oil.
12	do	8 ethylene oxide	6 stearic.
13	do	12 ethylene oxide	Do.
14	do	do	6 hydrogenated fatty acids from soya bean oil.
15	do	mixture of 3 ethylene oxide and 3 propylene oxide.	Do.
16	do	do	6 stearic.
17	do	3 propylene oxide	6 hydrogenated fatty acids from soya bean oil.
18	sorbitol	3 ethylene oxide <sup>1</sup>	Do.
19	mannitol	3 propylene oxide	Do.
20	pentaerythritol	4 ethylene oxide	4 stearic.
21	diglycerol	do	Do.
22	do	6 ethylene oxide	Do.
23	glycerol	3 ethylene oxide	3 stearic.
24	mannitol	6 ethylene oxide	6 palmitic.
25	glucose	5 ethylene oxide	4 stearic.
26	do	6 ethylene oxide	5 stearic.
27	erythritol	4 ethylene oxide	3 stearic.
28	mannitan	6 ethylene oxide	4 stearic.

<sup>1</sup> In No. 18 of the table the ethylene oxide condensation product of sorbitol was formed separately and then mixed with an equal proportion of the propylene oxide condensation product of mannitol after which the mixture was esterified.

These compounds may be applied singly or as a mixture to the filamentary material either in molten condition, in the form of a solution in a suitable organic solvent or in the form of a dispersion or emulsion in water. Where it is applied in the form of an emulsion, the temperature of the emulsion is preferably above the melting point of the compound. The filamentary material may be sprayed with or immersed in the solution of the compound. Where application is to be made to yarns and the like of continuous character, application may be made by directing them over a conventional form of roller running in a trough supplied with the solution or dispersion as the yarn runs from one package to another. Alternatively, a wound package of the yarn may be immersed in the dispersion or solution for a sufficient time to permit complete penetration. Excess solution is removed in any suitable manner, such as by a centrifugal hydro-extractor and then dried. The treated filamentary material may then be stored, if desired, under controlled humidity conditions until used in subsequent textile processing. The amount of the compound to be applied to the filamentary material depends upon the purpose for which it is applied and will be more fully explained hereinafter.

The compounds may be applied in conjunction with the application of other treating agents, such as antistatic agents, auxiliary softeners, lubricants, oils, or sizing agents. The auxiliary substances may be applied before or after the application of the compounds of the invention or they may be mixed and applied together in molten condition, or in the form of a dispersion, solution or emulsion.

The compounds of the invention are applicable to both natural and synthetic fibers or filaments. For example, they may be applied to fibers, filaments or yarns of cotton, wool, linen and silk, particularly silk which has been degummed, and other materials of natural origin. They are how-

ever of particular importance for application to synthetic textile materials, such as regenerated cellulose from viscose or cuprammonium solutions or to filamentary materials of organic derivatives of cellulose, such as cellulose acetate, of nylon, casein, and also the filamentary material derived

from the synthetic resins, such as the vinyl resins, particularly Vinyon.

These compounds may be applied to highly twisted yarns of rayon, such as to a 75 denier yarn having above 25 turns per inch, to reduce their harshness and stiffness and to set the twist therein. For the purpose of lubricating, imparting flexibility to and setting the twist in such yarn, to prepare them for subsequent textile processing, such as for the knitting of full fashioned hosiery or for weaving voile or crepe fabrics, the impregnation is controlled to incorporate within the yarn between about 8 to 11% of its weight of such compounds, preferably about 10%. For the application of this amount of the compound, a 50% concentration of such compound in an organic solvent, such as Stoddard solvent or other petroleum solvents such as that sold under the trade-mark Solvesso No. 3, is satisfactory. Alternatively, a 10% concentration of the compound in an aqueous dispersion may be employed. A dispersing agent, such as an alkyl aryl sulfonate, such as those sold under the trade name Nekal BX, may be employed to form the aqueous dispersion. During the severe flexing and rubbing action to which such treated yarns are subjected by the needles of knitting machines, substantially no stripping occurs and the knitted fabric has a firm but soft hand. After completion of the fabric, the conditioning compounds may be removed by a simple scouring operation. In the scouring, no harsh detergents needs be used in spite of the fact that the compounds are almost completely esterified with hydrophobic aliphatic groups.

For the lubricating of fibers or filaments in general, and in particular the harsh and stiff filaments of rayon, the application of the conditioning compounds is controlled so that from 1 to 5% or more by weight of the yarn is left thereon. In general the smaller percentage is sufficient to improve the feel and hand of the fibers and to render them pliant preparatory to spinning and felting.

Similarly for the purposes of lubricating yarns and rendering them pliant preparatory to twisting and winding, the lower percentage of the conditioning compound just specified may be sufficient in which case there is no point in applying a greater quantity.

For the purpose of lubricating and imparting flexibility to yarn which are to be woven or braided, it is preferably to impregnate the yarns with a larger proportion than is used in preparing yarns for knitting.

While preferred embodiments of the invention have been disclosed, 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 and scope of the invention as defined by the appended claims.

I claim:

1. Twisted textile filamentary material having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty acid ester of an ether condensation product of a polyol having at least three hydroxyl groups with an olefine oxide, the hydroxyl groups in the ether condensation product being substantially completely esterified, at least two thirds of the acid radicals in the ester being saturated, and the acid radicals therein having at least 16 carbon atoms in a straight chain.

2. Twisted yarns and the like having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty acid ester of an ether condensation product of a polyol having at least three hydroxyl groups with an olefine oxide having no more than 3 carbon atoms, the hydroxyl groups in the ether condensation product being substantially completely esterified, at least two thirds of the acid radicals in the ester being saturated, and the acid radicals therein having at least 16 carbon atoms in a straight chain.

3. Twisted yarns comprising fibers or filaments of regenerated cellulose having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty acid ester of an ether condensation product of a polyol having at least three hydroxyl groups with an olefine oxide having no more than 3 carbon atoms, the hydroxyl groups in the ether condensation product being substantially completely esterified, at least two thirds of the acid radicals in the ester being saturated, and the acid radicals therein having at least 16 carbon atoms in a straight chain.

4. Twisted yarns comprising fibers or filaments of regenerated cellulose having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty acid ester of a condensation product of a hexitol with an olefine oxide having no more than 3 carbon atoms, the hydroxyl groups in the condensation product being substantially completely esterified, at least two thirds of the acid radicals in the ester being saturated, and the acid radicals therein having at least 16 carbon atoms in a straight chain.

5. Twisted yarns and the like having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher saturated fatty acid ester of a condensation product of a hexitol with ethylene oxide, said product being substantially completely esterified, the fatty acid radicals therein having at least 16 carbon atoms in a straight chain.

6. Twisted yarns comprising fibers or filaments of regenerated cellulose having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty acid ester of a condensation product of a hexitol with ethylene oxide, the hydroxyl groups in the condensation product being substantially completely esterified, at least two thirds of the acid radicals in the ester being saturated, and the acid radicals therein having at least 16 carbon atoms in a straight chain.

7. Twisted yarns comprising fibers or filaments of regenerated cellulose having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty acid ester of a condensation product of sorbitol with an olefine oxide having no more than 3 carbon atoms, the hydroxyl groups in the condensation product being substantially completely esterified, at least two thirds of the acid radicals in the ester being saturated, the acid radicals therein having at least 16 carbon atoms in a straight chain.

8. Twisted synthetic yarns and the like having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher saturated fatty acid ester of a condensation product of sorbitol with ethylene oxide, said product being substantially completely esterified and the acid radicals therein having at least 16 carbon atoms in a straight chain.

9. Twisted yarns comprising fibers or filaments of regenerated cellulose having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty acid ester of a condensation product of sorbitol with ethylene oxide, the hydroxyl groups in the ether condensation product being substantially completely esterified, at least two thirds of the acid radicals in the ester being saturated, and the acid radicals therein having at least 16 carbon atoms in a straight chain.

10. Twisted yarns and the like having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a stearic acid ester of a condensation product of sorbitol with ethylene oxide, the hydroxyl groups of the condensation product being substantially completely esterified.

11. Twisted yarns comprising fibers or filaments of regenerated cellulose having the twist therein set with a compound comprising a stearic acid ester of a condensation product of about one mol of sorbitol with about six mols of ethylene oxide, said product being substantially completely esterified.

12. Twisted yarns and the like having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty acid ester of a condensation product of mannitol with ethylene oxide, the hydroxyl groups in the ether condensation product being substantially completely esterified, at least two thirds of the acid radicals in the ester being saturated, the acid radicals therein having at least 16 carbon atoms in a straight chain.

13. Twisted yarns comprising fibers or filaments of regenerated cellulose having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty ester of a condensation product of mannitol with ethylene oxide, the hydroxyl groups in the ether condensation product being substantially completely esterified, at least

7

two thirds of the acid radicals in the ester being saturated, and the acid radicals having at least 16 carbon atoms in a straight chain.

14. Twisted yarns and the like having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty acid ester of a condensation product of a hexose with an olefine oxide having no more than 3 carbon atoms, the hydroxyl groups in the ether condensation product being substantially completely esterified, at least two thirds of the acid radicals in the ester being saturated, and the acid radicals therein having at least 16 carbon atoms in a straight chain.

15. Twisted yarns and the like having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a higher fatty acid ester of a condensation product of glucose with ethylene oxide, the hydroxyl groups in the ether condensation product being substantially completely esterified, at least two thirds of the acid radicals in the ester being saturated, and the acid radicals therein having at least 16 carbon atoms in a straight chain.

16. Twisted yarns comprising fibers or fila-

8

ments of regenerated cellulose having the twist therein set with an unctuous solid having a melting point between about 30° C. and 70° C. comprising a stearic acid ester of a condensation product of glucose with about six mols of ethylene oxide, the hydroxyl groups in the ether condensation product being substantially completely esterified.

KENNETH R. BROWN.

#### REFERENCES CITED

The following references are of record in the file of this patent:

#### UNITED STATES PATENTS

Number	Name	Date
1,959,930	Schmidt et al. -----	May 22, 1934
1,970,578	Schoeller et al. -----	Aug. 21, 1934
2,297,135	Davis et al. -----	Sept. 29, 1942
20 Re. 20,417	Hood -----	June 22, 1937
2,207,229	Sherman -----	July 9, 1940

#### OTHER REFERENCES

Pub. "Spans and Tweens," Atlas Powder Co., Wilmington, Del., Nov. 1942.