Patented Jan. 10, 1939 2,143,765

UNITED STATES PATENT OFFICE

2,143,765

YARN CONDITIONING PROCESS AND COMPOSITION THEREFOR

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No Drawing. Application April 26, 1938, Serial No. 204,374

18 Claims. (Cl. 28—1)

This invention relates to the conditioning of textile yarns and more particularly to the conditioning of filaments and yarns composed of organic derivatives of cellulose such as cellulose acetate, cellulose propionate, cellulose acetate propionate, and cellulose acetate butyrate, to render them more amenable to textile operations such as knitting and the like.

As is well known in the manufacture of yarns, particularly those composed of or containing cellulose organic derivatives, it is necessary to treat the yarn in order to reduce the tendency toward breakage of the individual filaments or fibers when they are subjected to various mechanical strains and to lubricate the yarn in order to facilitate handling in such operations as spinning, twisting, winding and reeling. It is also necessary to treat yarn to adapt it for use as warp or filling or for the manufacture of various types of knitted fabrics. In knitting, it is particularly important that the yarn be soft and pliable in order that it may conform readily to the contour of the needles and thus produce a closely knit fabric free from such defects as "stitch distortion," "pin holes," "laddering," and the like.

Heretofore it has been proposed to employ softening agents such as polyhydric alcohols and similar agents as ingredients of yarn conditioning or lubricating formulas, generally in connection with mineral, animal or vegetable oils. It has been found, however, that most of the known softening agents and the various formulas containing them have certain drawbacks, one of the most serious of which is high vapor pressure, and in some cases too drastic a solvent action on the yarn. Many of such agents possess slight or insufficient solvent power for the lubricants with which they are used, and it is accordingly necessary to employ blending agents or emulsifying agents in order to obtain operable yarn treating formulas. In addition, many of the known softening and lubricating agents are insufficiently soluble in water to permit satisfactory removal by aqueous scour baths.

This invention has as its principal object to provide an entirely new class of yarn conditioning agents which are particularly adapted for the treatment of yarns composed of or containing organic derivatives of cellulose and capable of lubricating, softening and rendering such yarns more amenable to knitting and other textile operations. A further and specific object is to provide a class of conditioning agents which augment or assist the lubricating action of various lubricants when applied to such yarns. A still further object is to provide yarn softening and lubricating formulas which can be readily removed from the yarns by the usual scour baths. A still further object is to provide an improved method for the conditioning of yarns, particularly those composed of or containing organic derivatives of cellulose such as cellulose acetate, whereby the yarn is rendered soft and pliable and capable of employment in a variety of textile operations where complicated designs or stitches are employed. Another object is to provide an improved type of yarn which is especially amenable to textile operations including circular knitting, weaving, spinning and the like. Other objects will appear hereinafter.

These objects are accomplished by the following invention which, in its broader aspects, comprises the discovery that organic ester amides having the general formula:

$$(R'X)_a—N—(R)_a—\left(\begin{matrix}O\\ \|\\ O\end{matrix}C—R''\right)_b$$

where R is alkyl, cycloalkyl or aryl; R' and R'' are the same or different substituted or unsubstituted alkyl, aryl or cycloalkyl groups; X is —CO—; n is 1, b is 1 or more and R'X may stand for hydrogen, may be used as yarn conditioning agents and particularly as softening agents, with or without the addition of animal, mineral, or vegetable oils, in the treatment of yarns composed of or containing organic derivatives of cellulose. We have found that these compounds when employed as described in the detailed examples set forth below have a slight solvent and/or softening action on cellulose organic derivative yarns which renders such yarns soft and pliable without at the same time having too drastic an action thereon.

Typical examples of compounds which, in accordance with our invention, we have found to be valuable yarn-treating agents are given in the following table. These compounds which may, as indicated above, be considered ester amides, or more specifically as N,N-di(acyloxy-

alkyl) amides, are derived by reaction on alkylol amines of appropriate acids or anhydrides.

Ester amides derivable from monoethanolamine

Compound	Formula	Boiling range
		Millimeters
Diacetate	$CH_3CO—NH—C_2H_4OCOCH_3$	138–140° C/2
Acetate propionate	$CH_3CO—NH—C_2H_4OCOC_2H_5$	162–170° /18
Acetate propionate (isomer).	$C_2H_5CO—NH—C_2H_4OCOCH_3$	170–175° /19
Dipropionate	$C_2H_5CO—NH—C_2H_4OCOC_2H_5$	150–153° /5
Acetate butyrate	$CH_3CO—NH—C_2H_4OCOC_3H_7$	170–175° /19
Propionate butyrate	$C_2H_5CO—NH—C_2H_4OCOC_3H_7$	180–184° /20
Dibutyrate	$C_3H_7CO—NH—C_2H_4OCOC_3H_7$	188–192° /22
Butyrate caproate	$C_3H_7CO—NH—C_2H_4OCOC_5H_{11}$	200–210° /20
Dimethoxy acetate	$CH_3OCH_2CO—NH—C_2H_4OCOCH_2OCH_3$	205–210° /24

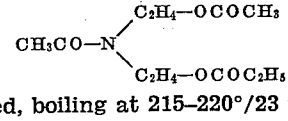
Ester amides derivable from diethanolamine

Compound	Formula	Boiling range
		Millimeters
Triacetate	$CH_3CO—N—(C_2H_4OCOCH_3)_2$	212–214° C/23
Diacetate propionate.	$C_2H_5CO—N—(C_2H_4OCOCH_3)_2$	220–°225 /23
Acetate dipropionate.	$CH_3CO—N—(C_2H_4OCOC_2H_5)_2$	215–220° /26
Diacetate butyrate	$C_3H_7CO—N—(C_2H_4OCOCH_3)_2$	220–225° /21
Tripropionate	$C_3H_7CO—N—(C_2H_4OCOC_2H_5)_2$	205–210° /20
Acetate dibutyrate	$CH_3—CO—N—(C_2H_4OCOC_3H_7)_2$	220–230° /23
Tributyrate	$C_3H_7CO—N—(C_2H_4OCOC_3H_7)_2$	220–222° /2
Acetate propionate butyrate.	$CH_3CO—N$ ($C_2H_4OCOC_2H_5$; $C_2H_4OCOC_3H_7$)	240–260° /24
Acetate butyrate caproate.	$CH_3CO—N$ ($C_2H_4OCOC_3H_7$; $C_2H_4OCOC_5H_{11}$)	232–240° /20
Dibutyrate propionate.	$C_2H_5CO—N—(C_2H_4OCOC_3H_7)_2$	222–230° /23
Dipropionate butyrate.	$C_3H_7CO—N—(C_2H_4OCOC_2H_5)_2$	218–225° /22
Tricaproate	$C_5H_{11}CO—N—(C_2H_4OCOC_5H_{11})_2$	235–245° /2

Examples of the preparation of typical compounds of this type are as follows:

Preparation of diethanolamine diacetate propionate

A mixture of 125 parts acetic anhydride and 80 parts propionic anhydride are added through a condenser to 60 parts diethanolamine. The reaction is extremely vigorous and a rate of addition is maintained so that the temperature remains at 100–110°. When all of the anhydrides have been added, the solution is refluxed gently for 30–60 minutes. The acids are then removed either at atmospheric pressure or under vacuum and the desired product distilled under diminished pressure. One hundred and fifty parts of



are obtained, boiling at 215–220°/23 mm.

Preparation of ethanolamine triacetate

Seven equivalents of acetyl chloride are added slowly to two equivalents of ethanolamine. The solution is then refluxed on the water bath until no more hydrogen chloride is given off. The product is then worked up in the usual way. A good yield of the triacetyl derivative is obtained, B. P. 165–170°/18 mm.

Preparation of diethanolamine tricaproate

One hundred and five parts of diethanolamine, 350 parts caproic acid, 5 parts concentrated sulfuric acid and 100 parts toluene are distilled in such a manner that the toluene is continuously separated from the water of reaction and returned to the reaction vessel. The distillation is stopped when 54 parts of water have been collected. The catalyst is then neutralized by washing with sodium carbonate solution and the product recovered by distillation. Three hundred and seventy-five parts of the tri-caproyl derivative are obtained, B. P. 235–245°/5 mm.

Preparation of ethanolamine propionate butyrate

Sixty one grams of ethanolamine, 130 grams of propionic anhydride and 160 grams of butyric anhydride are heated under reflux until the reaction is completed. The propionic and butyric acids formed in the reaction are distilled off and the ester-amide is distilled under reduced pressure. The compound is a colorless liquid and boils at 158–161°/11 mm.

In accordance with the invention these compounds may be applied directly to the yarn during or after spinning, or may be added to the spinning solution itself. We have found that these compounds have exceptional solvent powers which enable them to dissolve mineral oils and blown and unblown, drying and semi-drying, vegetable and animal oils and accordingly they may be, and preferably are, employed as ingredients of yarn conditioning or lubricating formulas in conjunction with agents which function wholly or partially as lubricants.

In the following examples and description we have set forth several of the preferred embodiments of our invention, but they are included merely for purposes of illustration and not as a limitation thereof.

Our invention will be more readily understood by reference to the following examples in which typical applications of the invention are set forth.

Example 1.—Yarn designed for knitting and composed of cellulose acetate is treated by applying thereto $(C_2H_5CO.OC_2H_4)_2N—CO—C_2H_5$ by means of an applicator roll. The amount of conditioning liquid may run from 4 to 25% by weight of the untreated yarn. If the yarn is intended for weaving, the same procedure may be followed, except that the amount of conditioning agent added to the yarn is only about 1 to 5%.

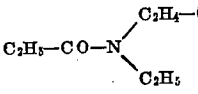
Example 2.—A conditioning liquid is made up by mixing the following ingredients in the indicated proportions:

$CH_3—CO—N—(C_2H_4—OCO—C_2H_5)_2$	80 parts
Olive oil	20 parts

This composition is applied to a cellulose acetate yarn intended for knitting in an amount representing 4 to 25% by weight of the untreated yarn. The filaments or fibers treated as described above are quite soft and pliable and give excellent results in textile operations, especially in circular knitting.

Other examples of yarn conditioning compositions which may be applied to various types of yarns, particularly those composed of or containing cellulose acetate, cellulose acetate propionate, cellulose acetate butyrate, and similar cellulose organic acid esters in accordance with our invention and which render such yarns soft and pliable and especially well adapted for various textile operations, particularly knitting, are as follows:

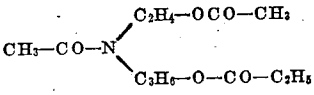
Example 3



80 parts

Blown olive oil 20 parts

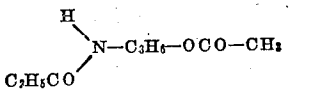
Example 4



20 parts

Light mineral oil 80 parts

Example 5



20 parts

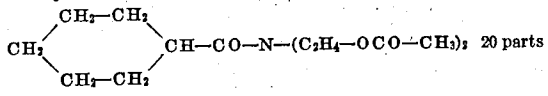
Blown sperm oil 80 parts

Example 6

$CH_3—OCH_2—CO—N—(C_3H_6OCO—CH_2—OCH_3)_2$ 40 parts

Neat's-foot oil 60 parts

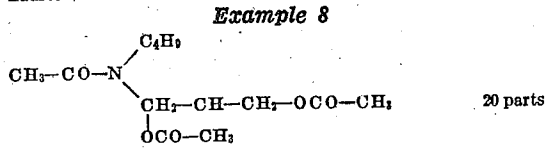
Example 7



20 parts

Sperm oil 60 parts
Laurol 20 parts

Example 8

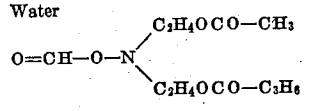


20 parts

Tetrahydrofurfuryl glycerol acetate 20 parts
Blown soya bean oil 60 parts

Example 9

Water 80 parts



10 parts

Water soluble cellulose ester 5 parts
Glycerol acetone 5 parts

Example 10

20% solution of cellulose acetate in acetone in which is incorporated 1–25% by weight of the cellulose acetate, of

$C_{17}H_{33}—CO—N—(C_2H_4—OCO—C_{17}H_{33})_2$,

is extruded through fine orifices into an evaporative atmosphere. The filaments thus produced are wound or twisted and wound. Yarns thus produced are pliable and suitable for knitting.

For the purpose of conditioning yarns composed of or containing various cellulose derivatives, particularly the cellulose organic acid esters such as cellulose acetate, cellulose propionate, cellulose acetate propionate, cellulose acetate butyrate and the like, we have found ethanolamine acetate propionate, ethanolamine dibutyrate, ethanolamine propionate butyrate, and diethanolamine tripropionate to be outstandingly effective. By the use of these compounds we are enabled to produce yarns or filaments having exceptional knitting properties. This is particularly true in the conditioning of yarns for use in circular knitting.

Conditioning liquids which we have found to be of particular value in preparing cellulose ester yarns for circular knitting are the following:

Example 11

	Parts
Blown neat's-foot oil	15
Ethanolamine propionate butyrate	85

Example 12

	Parts
Blown neat's-foot oil	20
Ethanolamine propionate butyrate	80

Example 13

	Parts
Blown olive oil	10–25
Ethanolamine propionate butyrate	90–75

Example 14

	Parts
Blown sperm oil	95–50
Ethanolamine propionate butyrate	5–50

Example 15

	Parts
Blown teaseed oil	5–50
Ethanolamine propionate butyrate	95–50

Example 16

	Parts
Ethanolamine propionate butyrate	100

Example 17

	Parts
Neat's-foot oil	5–50
Ethanolamine propionate butyrate	95–50

Example 18

	Parts
Blown neat's-foot oil	20
Ethanolamine dibutyrate	80

Example 19

	Parts
Blown neat's-foot oil	80
Diethanolamine tripropionate	20

Example 20

	Parts
Blown neat's-foot oil	80
Ethanolamine acetate propionate	20

Any of the above compositions may be applied to yarns intended for use in circular knitting by means of a bath, wick, spray, roller, pad or any suitable means. The amount of condition-

ing liquid applied may vary between 5–25% by weight of the yarn. Usually, however, the amount of conditioning liquid applied is about 10–15% by weight of the yarn. Yarn composed of cellulose acetate conditioned as described above gives excellent results when used in the circular knitting process.

As will be apparent from the above examples and description the conditioning agents of our invention may be applied by a wide variety of methods. For example, we may employ the agent as an ingredient of the spinning dope from which the filaments are formed, the amount of the agent so employed depending upon a number of factors, such as the particular cellulose derivative used in making the yarn, the solvent or solvent combination used in making up the spinning solution, and the degree of softness or pliability desired in the yarn, etc.

If the conditioning agent is to be applied to the yarn after spinning, this may be done by bringing the yarn in contact with a wick, roll, or felt wet therewith, or the liquid may be applied by immersion, spray, or otherwise. The particular point at which the liquid is applied may vary. It may, for example, be applied to the yarn inside or outside the spinning cabinet, between the guide and godet roll, between the godet or other roll or guide and the point of winding and/or twisting. In some cases, the liquid may even be applied to the yarn after winding onto cones, spools, bobbins, or the like or by the so-called "bobbin to bobbin" method. In the case of staple fiber manufacture, the liquid may be applied to the yarn prior to, or after cutting into staple lengths.

The amount of the agent so employed will vary widely depending upon the results desired, the specific nature of the material to which the agent is applied, the use to which the yarn is eventually to be put and other factors. For example, in a given case where a cellulose organic acid ester yarn such as a yarn composed of cellulose acetate, is intended for knitting, about 4 to 25% or more by weight, based on the weight of the dry yarn, may be satisfactory, while if the yarn is intended for weaving, the amount may vary between about 1 and 5%.

Although in the above examples we have referred primarily to yarn treating compositions containing only the conditioning agent and an oil, other ingredients such as solvents, non-solvents, emulsifying agents, blending agents and the like, may be added within the scope of our invention. Likewise, various dyes or other coloring matter may be included in case it is desired to permanently or fugitively tint or dye the material undergoing treatment.

Although we have found it convenient to illustrate our invention by reference to compositions containing specific percentages of the various ingredients, these percentages may vary widely depending upon the particular purpose for which the composition is intended. For example, if it is desired to control the solvent or softening action of the conditioning agent, the amount of the agent may be adjusted as, for example, by reducing the amount of the agent and correspondingly increasing the amount of oil or other ingredient.

While we have described our invention with particular reference to the treatment of yarns composed of organic derivatives of cellulose such as cellulose acetate, the conditioning agents and formulas described herein are applicable to the conditioning of many other types of cellulose derivative yarns such as those composed of or containing cellulose propionate, cellulose butyrate, cellulose acetate propionate, cellulose acetate butyrate, ethyl cellulose, methyl cellulose, benzyl cellulose and others, as well as to the conditioning of silk, wool, cotton, viscose and other natural or artificial materials.

The term "yarn" as used herein and in the claims is to be understood as including a single filament, a plurality of filaments associated into the form of a thread, either of high or low twist, single or multiple threads associated or twisted together, composite threads composed of a mixture of natural and artificial filaments or a composite thread formed by twisting together individual strands of natural or artificial materials, as well as cut staple fibers produced from natural and/or artificial filaments or threads and spun yarn produced from such staple fibers.

As indicated above, the yarn conditioning agents of our invention are exceptionally good solvents for a wide variety of mineral, blown and unblown, drying and semi-drying animal and vegetable oils such as cottonseed, olive, castor, neat's-foot, sperm and other oils. This enables them to be used with any of such oils in making up a variety of yarn treating formulas of varying composition.

The yarn conditioning method and compositions of our invention possess many outstanding advantages. The fundamental and outstanding characteristic of the agents employed in accordance with the invention is their ability to soften yarns, especially those composed of or containing organic derivatives of cellulose such as cellulose acetate and render them soft and pliable and amenable to various textile operations, especially operations such as those involved in weaving and knitting where complicated designs or stitches are employed, without too drastic an action on the yarn material. Another outstanding characteristic of these compounds is their exceptional solvent power for a wide variety of mineral, animal, and vegetable oils and their ability to act as lubricating assistants in conjunction with these oils when applied to such yarns. In addition, due to their solubility in water, they may be readily removed from yarns and fabrics by means of the usual aqueous scour baths. By employing the yarn conditioning agents and method of our invention as herein described, one is enabled to obtain highly satisfactory results in the manufacture of yarns and woven fabrics and especially the production from these yarns of closely knit fabrics free from defects such as pin holes, stitch distortion, laddering and the like.

What we claim is:

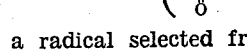
1. The process of conditioning yarn to render it more amenable to textile operations including knitting, weaving, spinning and the like which comprises applying thereto a lubricating and softening composition containing as its essential lubricating and softening component an organic ester amide having the general formula:

$$(R'X)_a\text{—}N\text{—}(R)_a\text{—}\left(\begin{matrix}O\\ \|\\ O\end{matrix}C\text{—}R''\right)_b$$

where R is a radical selected from the group consisting of alkyl, cycloalkyl and aryl; R′ and R″ are each a radical selected from the group consisting of substituted, and unsubstituted alkyl, aryl and cycloalkyl; X is —CO—; n is 1, b is at least 1 and R′X may be hydrogen.

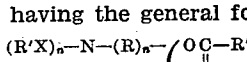
2. The process of conditioning yarn composed

of or containing organic derivatives of cellulose to render it more amenable to textile operations including knitting, weaving, spinning and the like which comprises applying thereto a lubricating and softening composition containing as its essential lubricating and softening compound an organic ester amide having the general formula:



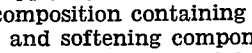
where R is a radical selected from the group consisting of alkyl, cycloalkyl and aryl; R′ and R″ are each a radical selected from the group consisting of substituted, and unsubstituted alkyl, aryl and cycloalkyl; X is —CO—; n is 1, b is at least 1 and R′X may be hydrogen.

3. The process of conditioning yarn composed of or containing cellulose acetate to render it more amenable to textile operations including knitting, weaving, spinning and the like, which comprises applying thereto a lubricating and softening composition containing as its essential lubricating and softening component an organic ester amide having the general formula:



wherein R is a radical selected from the group consisting of alkyl, cycloalkyl and aryl; R′ and R″ are each a radical selected from the group consisting of substituted, and unsubstituted alkyl, aryl and cycloalkyl; X is —CO—; n is 1, b is at least 1 and R′X may be hydrogen.

4. The process of conditioning yarn composed of or containing cellulose acetate to render it more amenable to textile operations including knitting, weaving, spinning and the like, which comprises applying thereto a lubricating and softening composition containing as its essential lubricating and softening component an organic ester amide having the general formula:



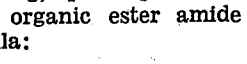
where R is a radical selected from the group consisting of alkyl, cycloalkyl and aryl; R′ and R″ are each a radical selected from the group consisting of substituted, and unsubstituted alkyl, aryl and cycloalkyl; X is —CO—; n is 1, b is at least 1 and R′X may be hydrogen, and textile lubricant.

5. The process of conditioning yarn composed of or containing cellulose acetate to render it more amenable to textile operations including knitting, weaving, spinning, and the like, which comprises applying thereto a lubricating and softening composition containing as its essential lubricating and softening component diethanolamine tripropionate.

6. The process of conditioning yarn composed of or containing cellulose acetate to render it more amenable to textile operations including knitting, weaving, spinning, and the like, which comprises applying thereto a lubricating and softening composition containing as its essential lubricating and softening component ethanolamine acetate propionate.

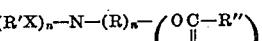
7. The process of conditioning yarn composed of or containing cellulose acetate to render it more amenable to textile operations including knitting, weaving, spinning and the like, which comprises applying thereto a lubricating and softening compositon containing as its essential lubricating and softening component ethanolamine propionate butyrate.

8. A conditioning agent for rendering yarns more amenable to textile operations including knitting, weaving, spinning and the like which comprises an organic ester amide having the general formula:



wherein R is a radical selected from the group consisting of alkyl, cycloalkyl and aryl; R′ and R″ are each a radical selected from the group consisting of substituted, and unsubstituted alkyl, aryl and cycloalkyl; X is —CO—; n is 1, b is at least 1 and R′X may be hydrogen.

9. A conditioning agent for rendering textile yarns composed of or containing organic derivatives of cellulose more amenable to textile operations including knitting, weaving, spinning and the like which comprises an organic ester amide having the general formula:



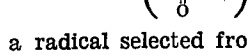
wherein R is a radical selected from the group consisting of alkyl, cycloalkyl and aryl; R′ and R″ are each a radical selected from the group consisting of substituted, and unsubstituted alkyl, aryl and cycloalkyl; X is —CO—; n is 1, b is at least 1 and R′X may be hydrogen and a textile lubricating oil.

10. A conditioning agent for rendering yarns composed of or containing cellulose acetate more amenable to textile operations including knitting, weaving, spinning, and the like containing diethanolamine tripropionate.

11. A conditioning agent for rendering textile yarns composed of or containing cellulose acetate more amenable to textile operations including knitting, weaving, spinning, and the like containing ethanolamine acetate propionate.

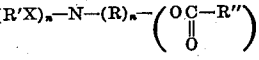
12. A conditioning agent for rendering textile yarns composed of or containing cellulose acetate more amenable to textile operations including knitting, weaving, spinning, and the like containing ethanolamine propionate butyrate.

13. Textile yarn amenable to textile operations including knitting, weaving, spinning and the like impregnated with a lubricant containing an organic ester amide having the general formula:



wherein R is a radical selected from the group consisting of alkyl, cycloalkyl and aryl; R′ and R″ are each a radical selected from the group consisting of substituted, and unsubstituted alkyl, aryl and cycloalkyl; X is —CO—; n is 1, b is at least 1 and R′X may be hydrogen.

14. Textile yarn composed of or containing organic derivatives of cellulose amenable to textile operations including knitting, weaving, spinning and the like, impregnated with a conditioning agent comprising an organic ester amide having the general formula:



wherein R is a radical selected from the group consisting of alkyl, cycloalkyl and aryl; R′ and R″ are each a radical selected from the group consisting of substituted, and unsubstituted alkyl, aryl and cycloalkyl; X is —CO—; n is 1, b is at least 1 and R′X may be hydrogen.

15. Textile yarn composed of or containing organic derivatives of cellulose amenable to tex-

tile operations including knitting, weaving, spinning and the like, impregnated with a conditioning agent comprising an organic ester amide having the general formula:

$$(R'X)_n—N—(R)_n—\left(\begin{matrix}O\\ \|\\ O\end{matrix}C—R''\right)_b$$

wherein R is a radical selected from the group consisting of alkyl, cycloalkyl and aryl; R' and R'' are each a radical selected from the group consisting of substituted, and unsubstituted alkyl, aryl and cycloalkyl; X is —CO—; *n* is 1, *b* is at least 1 and R'X may be hydrogen and a textile lubricant.

16. Textile yarns composed of or containing cellulose acetate amenable to textile operations including knitting, weaving, spinning and the like, impregnated with a lubricant comprising diethanolamine tripropionate.

17. Textile yarns composed of or containing cellulose acetate amenable to textile operations including knitting, weaving, spinning and the like, impregnated with a lubricant comprising ethanolamine acetate propionate.

18. Textile yarns composed of or containing cellulose acetate amenable to textile operations including knitting, weaving, spinning and the like, impregnated with a lubricant comprising ethanolamine propionate butyrate.

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