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UNITED STATES PATENT OFFICE

2,233,001

YARN CONDITIONING PROCESS

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7 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, deelectrifying, and otherwise 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 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, the manufacture of cut staple fibers, 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 phosphate and thiophosphate esters of hydroxy tertiary alkanol amines, 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. I 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.

I have found that the above-mentioned class of compounds can be prepared by treating a tertiary amine containing at least one hydroxyl group with suitable phosphating agents such as phopshoric acid, phosphorus pentaoxide and sulfide, phosphorus oxychloride or derivatives, phosphorus thiochloride and derivatives, etc.

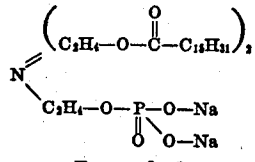
In addition to their use as yarn lubricants, I have discovered that these compounds may also be employed as wetting-out, dispersing and eulsifying agents, dye assistants, and as antistatic agents when applied to yarns particularly those composed of or containing organic derivatives of cellulose. Compounds of this type may be used in conjunction with older known textile lubricants such as blown and unblown teaseed, olive, sperm, neat's-foot, peanut, soya bean and mineral oils.

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

Example 1

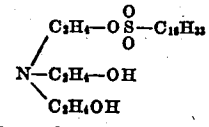
One mole of the dipalmityl ester of triethanolamine is dissolved in dioxane and six moles of pyridine are added. This solution is added dropwise to a solution of one mole of phosphorus oxychloride in dioxane. The reaction is completed by warming and most of the dioxane and pyridine is removed under reduced pressure. Then a cold solution of sodium carbonate is added. The reaction product is triethanolamine

dipalmitate phosphoric acid ester disodium salt and has the formula:

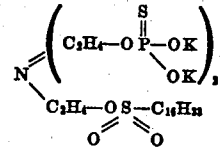


Example 2

One mole of triethanolamine monocetyl sulfonic acid ester having the formula:

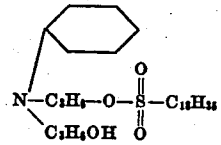


is reacted as described above with two moles of phosphorus thiochloride and the potassium salt prepared. The reaction product is trienthanolamine-bis-dipotassium thio-phosphate cetyl sulfonate ester and has the formula:

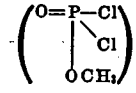


Example 3

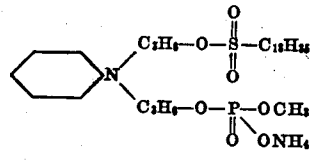
One mole of di-gamma-hydroxy propylaniline octadecyl sulfonate (prepared by treating di-gamma-hydroxypropylaniline with octadecane-sulfonylchloride in pyridine) and having the general formula:



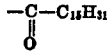
is reacted as described in Example 1 with one mole methoxy phosphonyl dichloride



and the ammonium salt, di-gamma-hydroxy propylaniline-octodecylsulfonate - a m m o n i u m phosphoric acid ester (monomethyl), prepared which has the formula



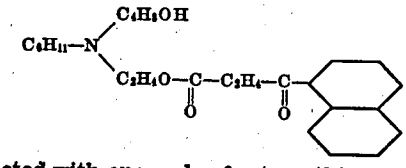
The phenyl group may contain a substituent such as —COOH, —SO_3H, —$C_{16}H_{33}$, —$OC_{18}H_{37}$,



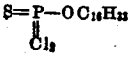
etc.

Example 4

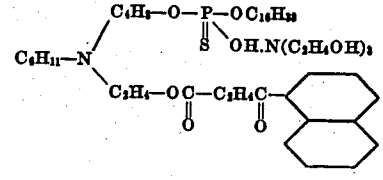
One mole of N-cyclohexyl-N-hydroxybutyl-N-hydroxy ethyl (B-α-naphthyl-keto propionate) having the formula



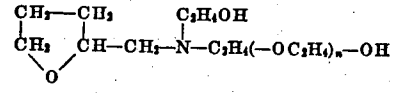
is reacted with one mole of cetoxy thiophosphory chloride having the formula:



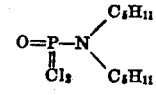
and the triethanolamine salt prepared by the same general technical method as described in Example 1. The reaction product is N-cyclohexyl-N-hydroxybutyl (cetoxy thiophosphato triethanolamine salt) - N - β - hydroxyethyl (β-α-naphthyl keto proprionate) and has the formula:



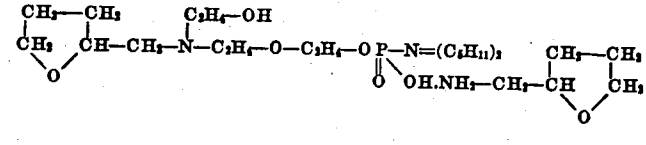
Example 5



One mole of the above compound, which is β-hydroxy ethyl - β - hydroxy-ethyl-β-ethoxy ethyl tetrahydrofurfurylamine is reacted as in Example 1 with one mole of diamyl phosphamidyl dichloride having the formula

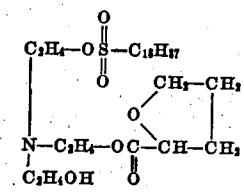


and the tetrahydrofurfurylamine salt prepared. The reaction product is β-hydroxyethyl-β-hydroxyethyl - β - ethyl ethyl tetrahydrofurfuryl-aminediamyl phosphamate ester tetrahydrofurfurylamine salt and has the formula:



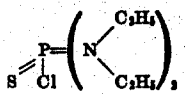
Example 6

One mole of triethanolamine-octadecyl sulfonate-tetrahydrofuroate having the formula

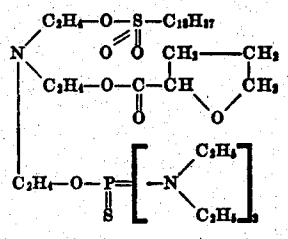


is reacted as in Example 1 with one mole of bisdi-

ethyl thiophosphamide monochloride having the formula

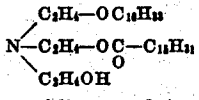


The reaction product is triethanolamine octadecyl sulfonate-tetra-hydrofuroate bisdiethyl thiophosphamate and has the formula:

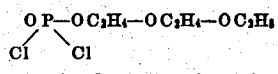


Example 7

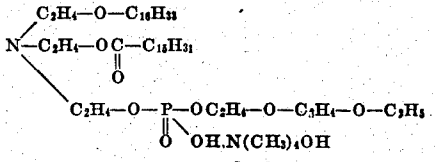
One mole of triethanolamine cetyl ether palmitic acid ester having the formula:



is dissolved in pyridine and treated as in Example 1 with one equivalent of β-ethoxy-β-ethoxy ethyl phosphoryl chloride having the formula:

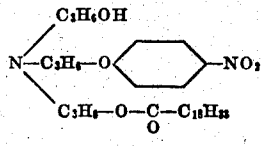


and the tetrametnyl ammonium hydroxide salt prepared. The reaction product is triethanolamine cetyl ether palmitic ester-β-ethoxy-β-ethoxy ethyl phosphate tetramethyl ammonium hydroxide salt and has the formula:

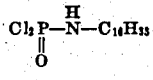


Example 8

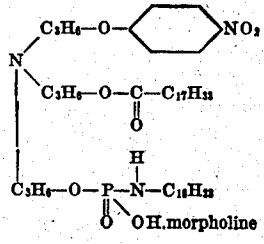
One mole of triethanolamine-p-nitrophenoxy ether-oleyl ester having the formula:



is reacted as in Example 1 with one mole of cetyl phosphamyl dichloride having the formula:



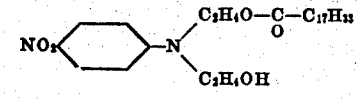
and the morpholine, pyridine, piperidine, etc. salt prepared. The reaction product is tripropanol-amine-p-nitro phenoxy-ether-oleyl ester cetyl phosphamyl ester morpholine salt and has the formula:



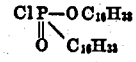
The nitro group may be reduced, diazotized and coupled to give oil-soluble dyes which are water soluble.

Example 9

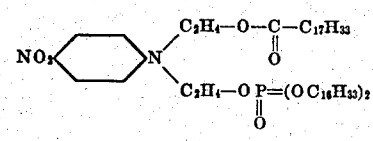
One mole of p-nitrodi-β-hydroxy ethylaniline oleate having the formula:



is reacted as in Example 1 with one mole of dicetoxy phosphoryl chloride having the formula:



The reaction product is p-nitrodi-β-hydroxy ethyl aniline-oleate phosphate dicetyl ester and has the formula:



The nitro group may be reduced, diazotized and coupled to give oil soluble dyes.

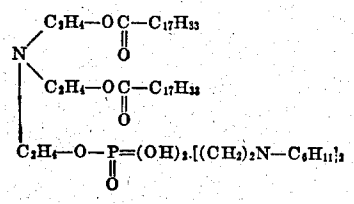
In accordance with the invention these compounds may be applied directly to the yarn during or after spinning, or may be added to the spinning solution itself. I have found that these compounds have exceptional solvent powers which enable them to dissolve mineral oils and blown and unblown, drying and 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, I have set forth several of the preferred embodiments of my invention, but they are included merely for purposes of illustration and not as a limitation thereof.

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

Example 10

Triethanolamine dioleate phosphate-dimethyl-cyclohexyl-amine salt having the formula:

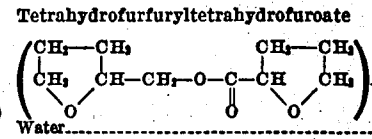


is applied to textile materials (silk, wool, cotton, viscose, cellulose acetate) by means of a bath, wick, roller, spray, etc. to facilitate their knitting, weaving, spinning and the like. Yarns lubricated with the above type of lubricant are of special value in the preparation of cut staple fibers. These fibers may be oiled before or after cutting.

Example 11

A conditioning liquid is made up as follows:

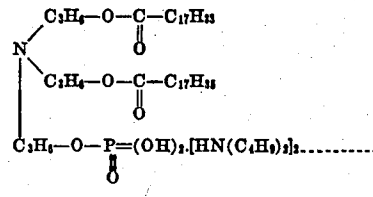
Tetrahydrofurfuryltetrahydrofuroate



Parts 30

Water 10

Tripropanolamine oleate stearate-phosphate dibutylamine salt



60

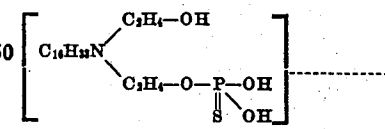
and applied to textile materials such as silk, wool, cotton, viscose, cellulose acetate, etc., as in Example 1. If the yarn is intended primarily for knitting the amount of conditioning liquid applied may vary from 4–25% by weight of the yarn, and if for weaving between 1–5% by weight. Cellulose acetate filaments treated as described above are quite soft and pliable and give improved results in various textile operations such as weaving, knitting, spinning, etc., and especially in the preparation of cut staple fibers.

Example 12

A conditioning liquid particularly useful in the preparation of cut staple fibers made of cellulose acetate and which may be used for other textile operations is made up as follows:

	Parts
Olive oil	99–90

Di-β-hydroxyethyl cetyl amine monothio phosphate

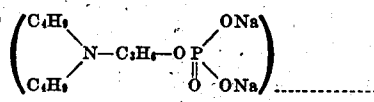


1–10

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 my invention and which render such yarns soft and pliable and especially well adapted for various textile operations, particularly knitting, and the manufacture of cut staple fibers, are as follows:

Example 13

N-N-dibutyl propanolamine-disodium phosphate

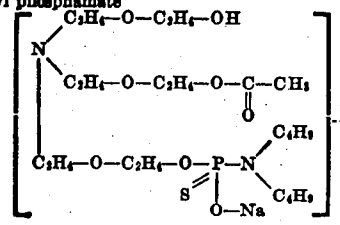


Parts 1–10

Blown olive oil	79–60
Ethanolamine propionate butyrate	20–30

Example 14

Tri [-β-hydroxy-β-ethoxy ethylamine acetate] sodium thiodibutyl phosphamate

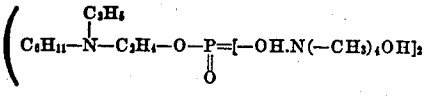


Parts 1–10

Neat's-foot oil	97–88
Sodium sulfate	2

Example 15

Ethyl-β-hydroxy ethyl cyclohexylamine phosphate (bis tetramethyl-ammonium hydroxide salt)



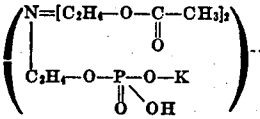
Parts 1–10

Blown neat's-foot oil	89–65
Oleic acid	10–25

Example 16

Sperm oil	49–20
Di-β-methoxyethyl succinate	30–50
Castor oil	20

Triethanolamine diacetate phosphate potassium salt

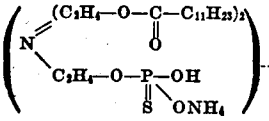


1–10

Example 17

Blown sperm oil	86–73
Sulfonated castor oil	10
Cetyl sulfate	1–5

Triethanolamine dilaurate thiophosphate ammonium salt



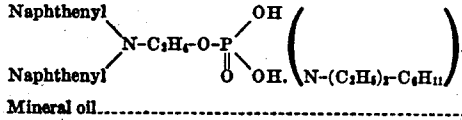
1–10

Diethylene glycol sodium sulfate 2

Example 18

Teaseed oil 89–80

Beta-hydroxyethyl dinaphthenylamine phosphate diethyl cyclohexylamine phosphate



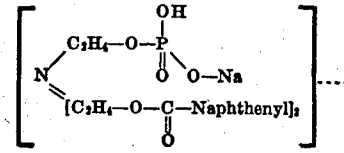
1–10

Mineral oil 10

Example 19

Blown teaseed oil	89–70
Castor oil	10–20

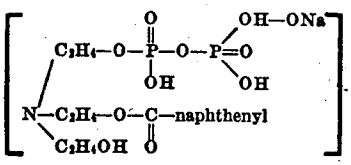
Triethanolamine dinaphthenate phosphate sodium salt



1–10

Example 20

Triethanolamine naphthenate pyrophosphate sodium salt

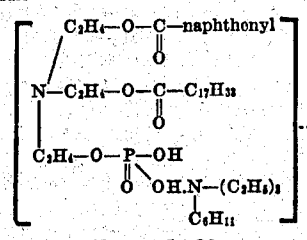


Parts 1–10

Mineral oil	10–20
Blown neat's-foot oil	20
Naphthenic acid	10
Blown sperm oil	59–40

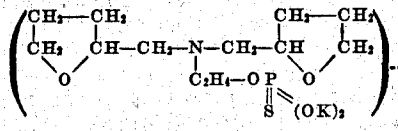
Example 21

	Parts
Light mineral oil	90
Triethanolamine naphthenate oleate phosphate diethylcyclohexylamine salt	



Example 22

	Parts
Water	75
Water soluble cellulose ester	10
Sulfonated castor oil	10
Beta-hydroxy ethyl ditetrahydrofurfurylamine thio phosphate dipotassium salt	



Any of the above compositions may be applied to the yarn intended for use in circular knitting by means of a bath, wick, spray, roller, pad or any suitable means. The amount of conditioning 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 my invention may be applied by a wide variety of methods. For example, I 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 I 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 my 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 I have found it convenient to illustrate my 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 I have described my 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 my 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 my invention possess many outstanding advantages. The fundamental and outstanding characteristic of the agents employed in accordance with the invention is their ability to de-electrify yarns, especially those composed of or containing organic derivatives of cellulose such as cellulose acetate and render them amenable to various textile operations, especially operations such as those involved in the manufacture of cut staple fibers and in weaving and knitting. 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 solubil-

ity 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 my 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 I claim is:

1. The process of conditioning yarn to render it more amenable to textile operations including knitting, weaving, spinning, the manufacture of staple fibers, and the like, which comprises applying a lubricating and anti-static composition containing as its essential lubricating and anti-static component an ester selected from the group consisting of phosphate and thiophosphate esters of partially esterified hydroxy tertiary alkanol amines.

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, the manufacture of staple fibers, and the like which comprises applying a lubricating and anti-static composition containing as its essential lubricating and anti-static component an ester selected from the group consisting of the phosphate and thiophosphate esters of partially esterified hydroxy tertiary alkanol amines.

3. The process of conditioning yarn composed of or containing cellulose acetate to render it more amenable to textile operations including knitting, weaving, spinning, the manufacture of staple fibers, and the like, which comprises applying a lubricating and anti-static composition containing as its essential lubricating and anti-static component an ester selected from the group consisting of phosphate and thiophosphate esters of partially esterified hydroxy tertiary alkanol amines.

4. The process of conditioning yarn composed of or containing cellulose acetate to render it more amenable to textile operations including knitting, weaving, spinning, the manufacture of staple fibers, and the like which comprises applying a lubricating and anti-static composition containing as its essential lubricating and anti-static component an ester selected from the group of phosphate and thiophosphate esters of partially esterified hydroxy tertiary alkanol amines and a textile lubricant.

5. Textile yarns amenable to textile operations including knitting, weaving, spinning, the manufacture of staple fibers, and the like impregnated with a lubricant and anti-static composition containing as its essential lubricating and anti-static component an ester selected from the group consisting of the phosphate and thiophosphate esters of partially esterified hydroxy tertiary alkanol amines.

6. Textile yarns composed of or containing organic derivatives of cellulose amenable to textile operations including knitting, weaving, spinning, the manufacture of staple fibers, and the like impregnated with a conditioning agent comprising an ester of phosphate and thiophosphate esters of partially esterified hydroxy tertiary alkanol amines.

7. Textile yarns composed of or containing organic derivatives of cellulose amenable to textile operations including knitting, weaving, spinning, the manufacture of staple fibers, and the like impregnated with a conditioning agent comprising an ester selected from the group consisting of phosphate and thiophosphate esters of partially esterified hydroxy tertiary alkanol amines, and a textile lubricant.

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