

UNITED STATES PATENT OFFICE

2,299,535

YARN CONDITIONING PROCESS AND COM-
POSITION THEREFORJoseph B. Dickey and James B. Normington,
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New JerseyNo Drawing. Application December 17, 1938,
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17 Claims. (Cl. 252-8.8)

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.

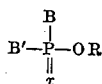
Heretofore it has been proposed to employ softening agents such as polyhydric alcohols and similar agents as ingredients of yarn conditioning or lubricating formulas, generally in connection with mineral, animal or vegetable oils. It has been found, however, that most of the known softening agents and 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 var-

ious 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 derivatives of phosphorus having the general formula:



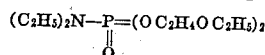
where x is a substituent selected from the group consisting of oxygen and sulfur; R is a substituent selected from the group consisting of alkyl and aryl groups, which groups may be substituted with other groups including alkoxy, polyalkoxy, tetrahydrofurfuryloxy, etc.: B is a substituent selected from the group consisting of and wherein B' is a substituted amino group, OR, -o alkyl, o-aryl and B', and wherein B' is a substituted amino group, 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.

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

PREPARATION OF COMPOUNDS

Example 1

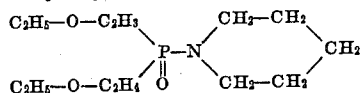
Di-(ethoxyethyl)-N,N-diethyl phosphonamide.
Diethylamino, di-(ethoxyethyl) phosphate.



Two equivalents of sodium are dissolved in an excess of ethoxyethanol and the solution cooled to 10°. One equivalent of diethylamino phosphoryl dichloride $[(C_2H_5)_2NPOCl_2]$ dissolved in ethoxyethanol, is then added slowly with vigorous agitation, keeping the temperature at 10°-15°. When all is added, the temperature is allowed to rise to that of the room and stirring is then continued for one hour. The product is filtered from the salt and fractionated. B. P. 167-70/10 mm.

Example 2

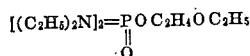
Di-(ethoxyethyl) phospho-piperidine.



One mole of piperidino-phosphoryl dichloride is added to two moles of potassium hydroxide dissolved in excess ethoxyethanol with agitation, at room temperature. The mixture is then heated on the steam bath for thirty minutes to complete the reaction, filtered and distilled. B. P. 210-13/22 mm.

Example 3

Ethoxyethyl-N,N,N',N'-tetraethyl phosphor-diamide.



To one equivalent of ethoxyethyl phosphoryl dichloride dissolved in 100 cc. ether are added four equivalents of diethylamine in 200 cc. ether, with stirring at 10°-15°. To complete the reaction, the mixture is refluxed for thirty minutes. The salt is then filtered out, and the filtrate distilled to obtain the desired product. B. P. 170-80/15 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 4

A conditioning liquid is made up as follows:

$CH_3-O-C_2H_4-O$	Parts
$\begin{array}{c} H \\ \\ P-N-C_2H_5 \\ \\ O \end{array}$	20
$CH_3-O-C_2H_4-O$	65
Olive oil.....	80

and applied to textile materials such as silk, wool, cellulose acetate, viscose, etc. as described above. If the yarn is intended for knitting primarily, the amount of conditioning liquid applied may vary from 4-25% by weight of the yarn, and if the yarn is intended for weaving, the amount of liquid applied may vary from 1-5% by weight of the yarn. Cellulose acetate filaments or fibers treated

as described are quite soft and pliable and give improved results in various operations such as weaving, knitting, etc.

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 5

$\begin{array}{c} OCH_3 \\ \\ C_2H_5OC_2H_4OC_2H_4O-P-N \\ \quad \\ S \quad C_2H_5 \end{array}$	Parts
Blown olive oil.....	75

Example 6

$\begin{array}{c} CH_2-CH_2 \\ \quad \\ (C_2H_5-O-C_2H_4-O-C_2H_4-O)_2-P-N \\ \quad \\ O \quad CH_2 \end{array}$	Parts
Light mineral oil.....	80

Example 7

$\begin{array}{c} H \\ \\ C_6H_{11}-O-C_2H_4-O-P-N-C_2H_4-O-C_2H_4-O-C_2H_5 \\ \quad \\ S \quad O-C_2H_5 \end{array}$	Parts
Neat's-foot oil.....	80

Example 8

$\begin{array}{c} C_{10}H_{21}-O-P-O-C_2H_4-O-CH_3 \\ \quad \\ S \quad N-C_2H_4 \\ \quad \\ C_2H_4 \quad O \end{array}$	Parts
Sperm oil.....	60
Laurel.....	20

Example 9

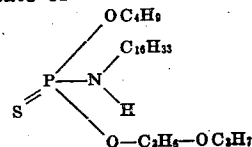
$\begin{array}{c} CH_2-CH_2 \\ \quad \\ P(=O)(OC_2H_4-OC_2H_4-O-CH_2-CH-CH_2) \\ \quad \quad \\ C_4H_9 \quad N \quad C_4H_9 \end{array}$	Parts
Tetrahydrofurfuryl alcohol.....	20
Blown soya bean oil.....	60

Example 10

Water.....	Parts
$(OC_2H_4-O-CH_3)_2$	80
$\begin{array}{c} P(=O) \\ \\ N-CH_2-CH_2 \\ \quad \\ C_2H_5 \quad CH_3 \end{array}$	10
Water soluble cellulose ester.....	5
Tetraethylene glycol.....	5

Example 11

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



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

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 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 ac-

tion 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.

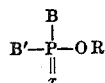
As indicated above, the yarn conditioning agents of our invention are exceptionally good solvents for a wide variety of mineral, blown and unblown, drying and semi-drying animal and vegetable oils such as cottonseed, olive, castor, neat's-foot, sperm and other oils. This enables them to be used with 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 and desire to secure by Letters Patent of the United States is:

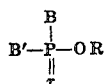
1. The process of conditioning yarn to render it more amenable to textile operations including knitting, weaving, spinning and the like, which comprises applying thereto a lubricating and softening composition containing as its essential

lubricating and softening component a derivative of phosphorus having the general formula:



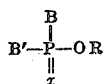
where x is a substituent selected from the group consisting of oxygen and sulfur; R is an alkoxy-alkyl group, B is a substituent selected from the group consisting of OR, -o-alkyl, -o-aryl and B'; and B' is a substituted amino group.

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 component a derivative of phosphorus having the general formula:



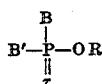
where x is a substituent selected from the group consisting of oxygen and sulfur; R is an alkoxy-alkyl group, B is a substituent selected from the group consisting of OR, -o-alkyl, -o-aryl and B'; and B' is a substituted amino group.

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 a derivative of phosphorus having the general formula:



where x is a substituent selected from the group consisting of oxygen and sulfur; R is an alkoxy-alkyl group, B is a substituent selected from the group consisting of OR, -o-alkyl, -o-aryl and B'; and B' is a substituted amino group.

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 a derivative of phosphorus having the general formula:



where x is a substituent selected from the group consisting of oxygen and sulfur; R is an alkoxy-alkyl group, B is a substituent selected from the group consisting of OR, -o-alkyl, -o-aryl and B'; and B' is a substituted amino group, and textile lubricant.

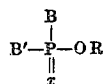
5. The process of conditioning yarn composed of or containing cellulose acetate to render it more amenable to textile operations including knitting, weaving, spinning, and the like, which comprises applying thereto a lubricating and softening composition containing as its essential lubricating and softening component dioctadecyl ester of di- β -methoxy ethyl phosphamate.

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 di- β -methoxy ethyl ester of monobutyl phosphamate.

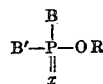
7. The process of conditioning yarn composed of or containing cellulose acetate to render it more amenable to textile operations including knitting, weaving, spinning, and the like which comprises applying thereto a lubricating and softening composition containing as its essential lubricating and softening component β' -ethoxy- β -ethoxyethyl-methyl ester of thio diethyl phosphamic acid.

8. Textile yarns amenable to textile operations including knitting, weaving, spinning, and the like, impregnated with a lubricating and softening composition containing as its essential lubricating and softening component a derivative of phosphorus having the general formula:



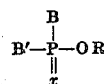
where x is a substituent selected from the group consisting of oxygen and sulfur; R is an alkoxy-alkyl group, B is a substituent selected from the group consisting of OR, -o-alkyl, -o-aryl and B'; and B' is a substituted amino group.

9. Textile yarns composed of or containing organic derivatives of cellulose amenable to textile operations including knitting, weaving, spinning, and the like impregnated with a lubricating and softening composition containing as its essential lubricating and softening component a derivative of phosphorus having the general formula:



where x is a substituent selected from the group consisting of oxygen and sulfur; R is an alkoxy-alkyl group, B is a substituent selected from the group consisting of OR, -o-alkyl, -o-aryl and B'; and B' is a substituted amino group.

10. Textile yarns composed of or containing cellulose acetate amenable to textile operations including knitting, weaving, spinning, and the like impregnated with a lubricating and softening composition containing as its essential lubricating and softening component a derivative of phosphorus having the general formula:



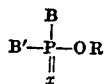
where x is a substituent selected from the group consisting of oxygen and sulfur; R is an alkoxy-alkyl group, B is a substituent selected from the group consisting of OR, -o-alkyl, -o-aryl and B'; and B' is a substituted amino group.

11. Textile yarns composed of or containing cellulose acetate amenable to textile operations including knitting, weaving, spinning, and the like, impregnated with a lubricant comprising dioctadecyl ester of di- β -methoxyethyl phosphamate.

12. Textile yarns composed of or containing cellulose acetate amenable to textile operations including knitting, weaving, spinning, and the like, impregnated with a lubricant comprising di- β -methoxy ethyl ester of monobutyl phosphamate.

13. Textile yarns composed of or containing cellulose acetate amenable to textile operations including knitting, weaving, spinning, and the like, impregnated with a lubricant comprising β' -ethoxy- β -ethoxyethyl-methyl ester of thio diethyl phosphamic acid.

14. A textile yarn lubricating and softening composition containing as its essential lubricating and softening agent a derivative of phosphorus having the general formula:



where x is a substituent selected from the group consisting of oxygen and sulfur; R is an alkoxy-alkyl group, B is a substituent selected from the

group consisting of OR, -o-alkyl, -o-aryl and B'; and B' is a substituted amino group.

15. A textile yarn lubricating and softening composition containing as its essential lubricating and softening agent dioctadecyl ester of di-beta-methoxyethyl phosphamate.

16. A textile yarn lubricating and softening composition containing as its essential lubricating and softening agent di-beta-methoxyethyl ester of monobutyl phosphamate.

17. A textile yarn lubricating and softening composition containing as its essential lubricating and softening agent β' -ethoxy-beta-ethoxyethyl-methyl ester of thio-diethyl phosphamic acid.

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