

UNITED STATES PATENT OFFICE

2,286,794

YARN CONDITIONING PROCESS AND
COMPOSITION THEREFOR

Joseph B. Dickey and James G. McNally, Rochester, N. Y., assignors to Eastman Kodak Company, Rochester, N. Y., a corporation of New Jersey

No Drawing. Application October 9, 1940,
Serial No. 360,451

18 Claims. (Cl. 252-8.8)

This invention relates to the treatment of cellulose organic derivative filaments, fibers, threads and the like and more particularly to a method of eliminating the tendency of such materials to accumulate charges of static electricity in twisting, winding and spinning. The invention also relates particularly to the treatment of cellulose organic derivative cut staple fibers to adapt them for the manufacture of spun yarns.

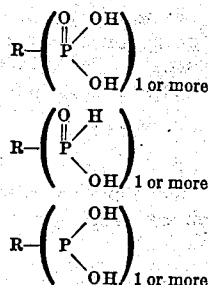
As is well known, cellulose organic derivative materials, particularly in filament, yarn, or sheet form, are particularly subject to the accumulation on their surfaces of charges of static electricity from frictional and other influences. In the case of filaments and yarns in which the surface area of the material (per unit of weight) is enormously greater as compared to sheets or films, the problem of static often assumes serious proportions. In the manufacture of yarns of various types from artificial filaments, numerous steps in which the yarn is subjected to frictional influences may be necessary, as for example, twisting, winding, warping, carding, combing, drawing, spinning and the like, all of which operations tend to produce large accumulations of static on the filaments. This is particularly aggravated in the utilization of cut staple, as for example, in the making of spun yarn, since in carding and combing considerable friction is developed and as a result sufficient charges accumulate on the fibers to make the formation of the web difficult, if not impossible; furthermore, even if a web is formed with some degree of success, the subsequent drafting operation produces a very uneven spun yarn or intermediate roving due to the presence of the static charge. In addition, there is the ever-present fire hazard.

Cellulose derivative yarn, particularly in the form of cut staple, is extremely difficult to handle in the various operations involved in making spun yarn, not only because such material is composed of filaments having smooth, glass-like surfaces without natural kink or irregularity, but also because it has relatively negligible moisture content at normal humidities, as compared to natural fibers such as cotton and wool, making it inherently far more susceptible to the accumulation of static. The charges accumulating on cellulose derivative cut staple fibers are, under some circumstances, even sufficient to separate the individual fibers a distance of inches, notwithstanding their relatively minute size.

This invention has as its principal object to provide an improved method for the treatment of cellulose organic derivative yarns to reduce

or eliminate the tendency of such yarns to accumulate charges of electricity under frictional and other influences. A further object is to provide a method of facilitating the handling and employment of cellulose organic derivative cut staple fibers in the various operations involved in the manufacture of spun yarns. A still further object is to provide a method of treating cellulose organic derivative yarns in either filament or cut staple form, wherein the material is simultaneously softened and lubricated and the accumulation of static charges thereon is prevented. Another object is to provide an improved type of cellulose organic derivative cut staple having little or no tendency toward accumulation of static electrical charges. Other objects will appear hereinafter.

These objects are accomplished by the following invention which in its broader aspects comprises applying to cellulose organic derivative yarns either in the form of filaments, threads or stable fibers, an antistatic composition characterized by the presence therein of a salt of an acid of phosphorus selected from the group consisting of the phosphonic and phosphinic acids. The compounds from which these salts are prepared may be said to have the following structural formula:



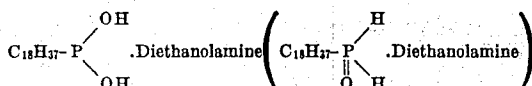
wherein R is an alkyl group. The phosphonic and phosphinic acids and their preparation have been described in general in U. S. Patent 2,137,792. In preparing salts the acids are reacted with one or two equivalents of any desired organic or inorganic base.

In accordance with our invention the above-mentioned salts may be applied to the yarn in any convenient manner, for example, by roll, wick, immersion, spraying or the like. Inasmuch as these compounds are soluble in oil they may be dissolved or dispersed in oils such as mineral oil, blown and unblown, drying, and semi-drying, vegetable and animal oils, examples of which are light mineral, neat's-foot, olive, teaseed, castor,

soya bean, rapeseed oils, etc., and other 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.

Example 1

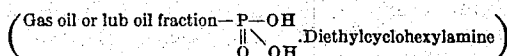


is applied to textile fibers or threads by means of a bath, wick, roller, spray, etc. to facilitate their knitting, weaving, spinning and the like. Yarns lubricated (1-25%) with compounds of this type are of special value in the preparation of cut staple fibers. These fibers may be oiled before or after cutting.

Example 2

A conditioning liquid is made up as follows:

70 parts glycerol acetone butyrate
30 parts diethylcyclohexylamine salt of the phosphonic acids derived from gas oil or lubricating oil fractions



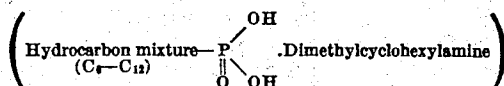
and applied to textile materials (silk, wool, cotton, viscose, cellulose derivatives, etc.) as in Example 1. If the yarn is intended primarily for knitting, the amount of conditioning liquid applied may vary from 4-25% by weight of the yarn and if for weaving, between 1-5% by weight of the yarn.

Cellulose acetate filaments treated as described above are quite soft and pliable and give improved results in various textile operations such as weaving, knitting, etc., and especially in the production of cut staple fibers.

Conditioning liquids particularly useful for the treatment of various types of yarns in accordance with our invention particularly those composed of or containing cellulose acetate, cellulose acetate propionate, cellulose acetate butyrate, and similar organic acid esters, and especially adapted for the deelectrification or anti-static treatment of cellulose organic derivative cut staple yarns are as follows:

Example 3

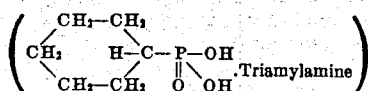
40 parts olive oil
1-10 parts dimethylcyclohexylamine salt from the phosphonates from a mixture of C_8 to C_{12} hydrocarbons



59-50 parts ethanolaminepropionatebutyrate

Example 4

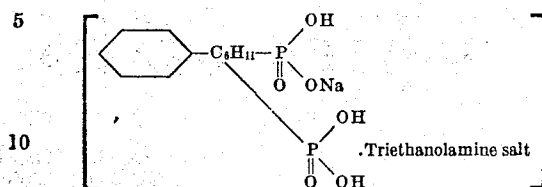
1-10 parts cyclohexanephosphonate triamylamine salt



99-90 parts blown olive oil

Example 5

1-5 parts hexylbenzene diphosphonate sodium-triethanolamine salt



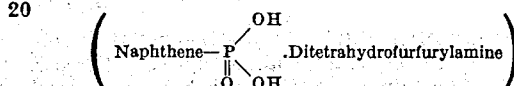
2 parts sodium sulfate

92-78 parts neat's-foot oil

15 5-15 parts naphthenic acid

Example 6

1-10 parts naphthene phosphonate ditetrahydrofurfurylamine salt



79-50 parts blown neat's-foot oil

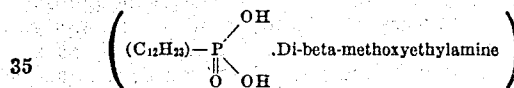
25 20-40 parts tetrahydrofurfuryl formal

Example 7

85 parts blown teaseed oil

10 parts oleic acid

30 5 parts hexylcyclohexane phosphonate-di-beta-methoxyethylamine salt

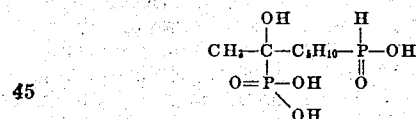


Example 8

97-88 parts sperm oil

40 2 parts ammonium phosphate

1-10 parts



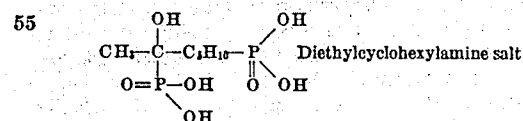
(Beta-hydroxy-beta-phosphonic heptane phosphonic acid cyclohexylamine salt)

Example 9

89-70 parts blown sperm oil

10-20 parts oleic acid

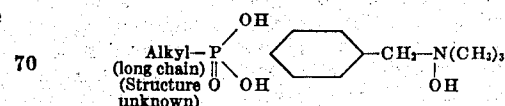
1-10 parts



60 (Beta-hydroxy-beta-phosphonic hexane phosphonic acid diethylcyclohexylamine salt)

Example 10

65 5 parts (benzyl trimethyl ammonium hydroxide salt of the alkyl phosphonic acid of polymeric hydrocarbons from cracked gasoline)



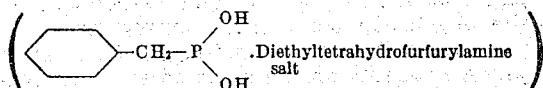
55 parts blown olive oil

20 parts blown neat's-foot oil

75 20 parts tetrahydrofurfuryl formal

Example 11

5 parts benzylphosphinate diethyltetrahydrofurfurylamine salt



15 parts sulfonated castor oil

2 parts water

3 parts glycerol

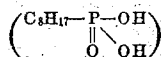
75 parts olive oil

Example 12

5 parts water soluble cellulose ester

85 parts water

5 parts octyl phosphonate



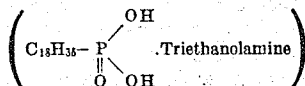
5 parts sulfonated castor oil

Example 13

85 parts water

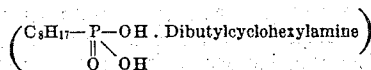
10 parts gelatin

5 parts oleyl phosphonate triethanolamine salt

*Example 14*

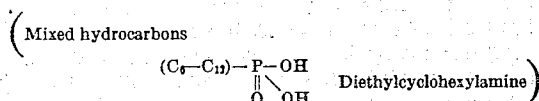
99-90 parts light mineral oil

1-10 parts octylphosphonate dibutylcyclohexylamine salt

*Example 15*

99-90 parts butyl stearate

1-10 parts mixed hydrocarbon (6-12) phosphonate diethylcyclohexylamine salt

*Example 16*

In a 20% acetone solution of cellulose acetate is dissolved 1-5% by weight of benzyl trimethylammonium hydroxide salt of the alkyl phosphonic acid of polymeric hydrocarbons from cracked gasoline. The acetone solution is then extruded into an evaporative atmosphere and the filaments thus produced are wound, or twisted and wound. If desired, the filaments produced by this method are oiled with any of the combinations of lubricants or lubricants plus conditioning agents previously described. Filaments thus prepared are of particular value in the preparation of cut staple fibers.

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 agents 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 properties desired in the finished 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 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 deelectrifying 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 deelectrify 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, as well as 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 solubility in water, they may be readily removed from yarns and fabrics by means of the usual aqueous scour baths.

Of the above compositions we have found of particular value the dialkyl cyclohexylamine salts of phosphonic acids derived from petroleum and gas oil hydrocarbons. As will be understood, these hydrocarbons cannot be identified by reference to any specific formula, since a large number of compounds are present in any petroleum or gas oil fraction. In general, however, it may be said that such hydrocarbons can be regarded to contain at least six carbon atoms and which may contain one or more double bonds. Of these dialkyl cyclohexylamine salts we have found as of particular value the diethyl cyclohexylamine salt of the alkyl phosphonic acids derived from such hydrocarbons, the dimethyl cyclohexylamine salt obtained from the phosphonates from a mixture of C₆-C₁₂ hydrocarbons and the benzyl trimethyl ammonium hydroxide salt of the alkyl phosphonic acid of polymeric hydrocarbons from cracked gasoline.

What we claim and desire to secure by Letters Patent of the United States is:

1. The method of eliminating the tendency of yarns, filaments or fibers to accumulate charges of static electricity, which comprises applying thereto a composition containing as its essential anti-static component a salt of an acid of phosphorus selected from the group consisting of the alkyl phosphonic and phosphinic acids in which the alpha carbon atom of the alkyl group is devoid of hydroxyl groups.

2. The method of eliminating the tendency of yarns, filaments or fibers composed of or containing organic derivatives of cellulose to accumulate charges of static electricity, which com-

prises applying thereto a composition containing as its essential anti-static component a salt of an acid of phosphorus selected from the group consisting of the alkyl phosphonic and phosphinic acids in which the alpha carbon atom of the alkyl group is devoid of hydroxyl groups.

3. The method of eliminating the tendency of yarns, filaments or fibers composed of or containing cellulose acetate to accumulate charges of static electricity, which comprises applying thereto a composition containing as its essential anti-static component a salt of an acid of phosphorus selected from the group consisting of the alkyl phosphonic and phosphinic acids in which the alpha carbon atom of the alkyl group is devoid of hydroxyl groups.

4. The method of eliminating the tendency of yarns, filaments or fibers composed of or containing organic derivatives of cellulose, to accumulate charges of static electricity, which comprises applying thereto a composition containing as its essential anti-static component diethylcyclohexylamine salt of the alkyl phosphonic acids derived from gas oil or lubricating oil fractions.

5. The method of eliminating the tendency of yarns, filaments or fibers composed of or containing organic derivatives of cellulose to accumulate charges of static electricity, which comprises applying thereto a composition containing as its essential anti-static component dimethylcyclohexylamine salt from the phosphonates from a mixture of C₆ to C₁₂ hydrocarbons.

6. The method of eliminating the tendency of yarns, filaments or fibers composed of or containing organic derivatives of cellulose to accumulate charges of static electricity, which comprises applying thereto a composition containing as its essential anti-static component benzyl trimethyl ammonium hydroxide salt of the alkyl phosphonic acid of polymeric hydrocarbons from cracked gasoline.

7. A composition adapted, when applied to textile yarns, filaments or fibers to eliminate the tendency of such materials to accumulate charges of static electricity, said composition containing as its essential anti-static component a salt of an acid of phosphorus selected from the group consisting of the alkyl phosphonic and phosphinic acids in which the alpha carbon atom of the alkyl group is devoid of hydroxyl groups.

8. A composition adapted, when applied to cellulose organic derivative yarns, filaments or fibers, to eliminate the tendency of such materials to accumulate charges of static electricity, said composition containing as its essential anti-static component a salt of an acid of phosphorus selected from the group consisting of the alkyl phosphonic and phosphinic acids in which the alpha carbon atom of the alkyl group is devoid of hydroxyl groups.

9. A composition adapted, when applied to cellulose acetate yarns, filaments or fibers to eliminate the tendency of such materials to accumulate charges of static electricity, said composition containing as its essential anti-static component a salt of an acid of phosphorus selected from the group consisting of the alkyl phosphonic and phosphinic acids in which the alpha carbon atom of the alkyl group is devoid of hydroxyl groups.

10. A composition adapted, when applied to cellulose organic derivative yarns, filaments or fibers, to eliminate the tendency of such materials to accumulate charges of static electricity, said composition containing as its essential anti-

static component diethylcyclohexylamine salt of the alkyl phosphonic acids derived from gas oil or lubricating oil fractions.

11. A composition adapted, when applied to cellulose organic derivative yarns, filaments or fibers, to eliminate the tendency of such materials to accumulate charges of static electricity, said composition containing as its essential anti-static component dimethylcyclohexylamine salt from the phosphonates from a mixture of C₆ to C₁₂ hydrocarbons.

12. A composition adapted, when applied to cellulose organic derivative yarns, filaments, or fibers, to eliminate the tendency of such materials to accumulate charges of static electricity, said composition containing as its essential anti-static component benzyl trimethyl ammonium hydroxide salt of the alkyl phosphonic acid of polymeric hydrocarbons from cracked gasoline.

13. Textile yarns having a lessened tendency to accumulate charges of static electricity, said yarns containing a salt of an acid of phosphorus selected from the group consisting of the alkyl phosphonic and phosphinic acids in which the alpha carbon atom of the alkyl group is devoid of hydroxyl groups.

14. Textile yarns composed of or containing organic derivatives of cellulose having a lessened tendency to accumulate charges of static electricity, said yarns containing a salt of an acid of phosphorus selected from the group consisting of the alkyl phosphonic and phosphinic acids in

which the alpha carbon atom of the alkyl group is devoid of hydroxyl groups.

15. Textile yarns composed of or containing cellulose acetate having a lessened tendency to accumulate charges of static electricity, said yarns containing a salt of an acid of phosphorus selected from the group consisting of the alkyl phosphonic and phosphinic acids in which the alpha carbon atom of the alkyl group is devoid of hydroxyl groups.

16. Textile yarns composed of or containing organic derivatives of cellulose having a lessened tendency to accumulate charges of static electricity, said yarns containing diethylcyclohexylamine salt of the alkyl phosphonic acids derived from gas oil or lubricating oil fractions.

17. Textile yarns composed of or containing organic derivatives of cellulose having a lessened tendency to accumulate charges of static electricity, said yarns containing dimethylcyclohexylamine salt from the phosphonates from a mixture of C₆ to C₁₂ hydrocarbons.

18. Textile yarns composed of or containing organic derivatives of cellulose having a lessened tendency to accumulate charges of static electricity, said yarns containing benzyl trimethyl ammonium hydroxide salt of the alkyl phosphonic acid of polymeric hydrocarbons from cracked gasoline.

JOSEPH B. DICKEY.
JAMES G. McNALLY.