

UNITED STATES PATENT OFFICE

2,279,502

YARN CONDITIONING PROCESS AND
COMPOSITION THEREFOR

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No Drawing. Application October 9, 1940,
Serial No. 360,454

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.

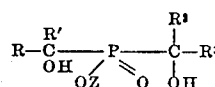
As is well known, cellulose organic derivative materials, particularly in filament, yarn, or sheet form, are particularly subject to the accumulation on their surfaces of charges of static electricity from frictional and other influences. In the case of filaments and yarns in which the surface area of 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 staple fibers, an anti-static composition characterized by the presence therein of salts of dialkyl phosphonic acids, said acids having the general formula:



wherein R' and R² are substituents selected from the group consisting of hydrogen and alkyl and R and R³ are substituents selected from the group consisting of alkyl, cycloalkyl, aryl and heterocyclic groups and hydrogen, and Z is hydrogen or a salt-forming radical, and wherein R, R', R², and R³ may be part of a cyclic system of carbon atoms. The alkyl group may be selected from methyl, ethyl, butyl, cycloalkyl, benzyl, cetyl, ethoxyethyl, allyl and tetrahydrofurfuryl and the like. The aryl group may be phenyl, zenyl, naphthyl and furyl, etc.

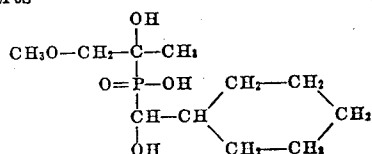
These compounds may be prepared as described in Compt. rend., 134,847; Ann. Chim. Phys. 8, 3,347; Compt. rend., 133,219.

In accordance with our invention the above-mentioned salts of dialkyl phosphonic acids 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, neats-foot, olive, teaseed, castor, soya bean, rapeseed oils, 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 11

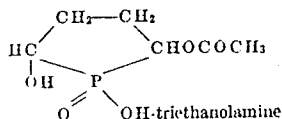
Teaseed oil, 99-90 parts
1-10 parts



(Beta-hydroxy-alpha-methoxy propyl-omega-hydroxy-hexahydro-benzyl phosphonic acid)

Example 12

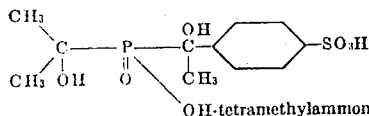
Glycerolacetone acetate, 60-70 parts
Blown teaseed oil 39-20 parts
1-10 parts



(Alpha-hydroxy-alpha-acetoxy cyclobutyl-phosphonic acid triethanolamine salt)

Example 13

1-10 parts

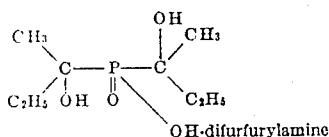


(Beta-hydroxy propyl-beta-hydroxy-beta-phenyl (p sulfonic acid) phosphonic acid tetramethylammonium hydroxide salt)

Blown olive oil, 30 parts
Blown neat's-foot oil, 69-60 parts

Example 14

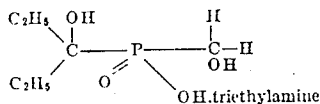
Olive oil, 55-50 parts
Sulfonated castor oil, 10 parts
Mineral oil, 20 parts
Oleic acid, 10-15 parts
5 parts



(Bis-beta-hydroxybutane phosphonic acid difurfurylamine salt)

Example 15

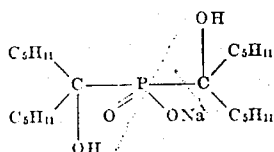
70 parts water
15 parts water soluble cellulose ester
10 parts sulfonated olive oil
5 parts



(Gamma-hydroxy pentane-alpha-hydroxy methane phosphonic acid triethylamine salt)

Example 16

One pound 1-methylamino-4-ethylamino-anthraquinone is ground with 1/4 pound

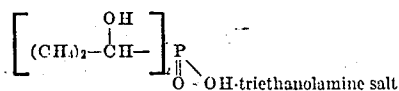


(Bis-6-hydroxy undecyl phosphonic acid sodium salt)

and 3 pounds of sugar. The resulting finely divided mixture is then added to 100 gallons of water heated to 80-90°. The resulting aqueous suspension of blue dye is then used to dye about 100 pounds of cellulose acetate deep blue shades.

Example 17

One pound of o-ethoxybenzene azo barbituric acid is ground with 1/2 pound of



(Bis-beta-hydroxy propane phosphonic acid triethanolamine salt)

and 3 pounds of sugar as in Example 16. The resulting finely divided mixture is poured into water (100 gallons) heated to 80-85° and used to dye 100 pounds of cellulose acetate greenish-yellow shades in any desired manner.

As will be evident from a consideration of Examples 16 and 17, the conditioning agents of our invention may be used effectively as dispersing agents for dyes. These agents also have exceptional value in other relationships in which emulsifying and dispersing power are important. In other words, they may be employed for a wide variety of uses in the textile industry.

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.

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 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.

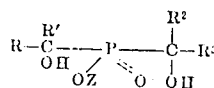
The yarn conditioning method and compositions of our invention possess many outstanding advantages. The fundamental and outstanding characteristic of the agents employed in accordance with the invention is their ability to deelectrify yarns, especially those composed of or containing organic derivatives 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 the manufacture of yarns adapted for 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 that bis-beta-hydroxy propane-beta-phosphonic acid diethanolamine salt, bis-beta-hydroxy butane-beta-phosphonic acid diethylcyclohexylamine

salt, and bis-beta-hydroxy pentane-beta-phosphonic acid ammonium salt are outstanding in their ability to reduce or eliminate the tendency of yarns composed of or containing organic derivatives of cellulose to accumulate charges of static electricity.

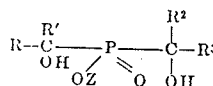
What we claim and desire to secure by Letters Patent of the United States is:

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 a dialkyl phosphonic acid having the general formula:



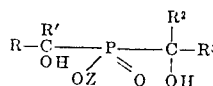
- wherein R' and R² are substituents selected from the group consisting of hydrogen and alkyl and R and R³ are substituents selected from the group consisting of alkyl, cycloalkyl, and heterocyclic groups and hydrogen, and Z is a substituent selected from the group consisting of hydrogen and salt-forming radicals.

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 comprises applying thereto a composition containing as its essential anti-static component a salt of a dialkyl phosphonic acid having the general formula:



- wherein R' and R² are substituents selected from the group consisting of hydrogen and alkyl and R and R³ are substituents selected from the group consisting of alkyl, cycloalkyl, and heterocyclic groups and hydrogen, and Z is a substituent selected from the group consisting of hydrogen and salt-forming radicals.

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 a dialkyl phosphonic acid having the general formula:



- wherein R' and R² are substituents selected from the group consisting of hydrogen and alkyl and R and R³ are substituents selected from the group consisting of alkyl, cycloalkyl, and heterocyclic groups and hydrogen, and Z is a substituent selected from the group consisting of hydrogen and salt-forming radicals.

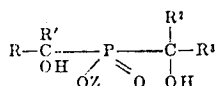
4. The method of eliminating the tendency of yarns, filaments or fibers composed of or containing organic derivatives of cellulose to accumulate charges of static electricity, which comprises applying thereto a composition containing as its essential anti-static component bis-beta-hydroxy propane-beta-phosphonic acid diethanolamine salt.

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 bis-beta-hydroxy butane-beta-phosphonic acid diethylcyclohexylamine salt.

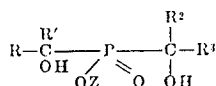
6. The method of eliminating the tendency of yarns, filaments or fibers composed of or containing organic derivatives of cellulose to accumulate charges of static electricity, which comprises applying thereto a composition containing as its essential anti-static component bis-beta-hydroxy pentane-beta-phosphonic acid ammonium salt.

7. An anti-static 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 a dialkyl phosphonic acid having the general formula:



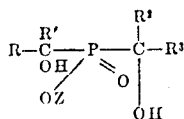
wherein R' and R² are substituents selected from the group consisting of hydrogen and alkyl and R and R³ are substituents selected from the group consisting of alkyl, cycloalkyl, and heterocyclic groups and hydrogen, and Z is a substituent selected from the group consisting of hydrogen and salt-forming radicals.

8. An anti-static 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 a dialkyl phosphonic acid having the general formula:



wherein R' and R² are substituents selected from the group consisting of hydrogen and alkyl and R and R³ are substituents selected from the group consisting of alkyl, cycloalkyl, and heterocyclic groups and hydrogen, and Z is a substituent selected from the group consisting of hydrogen and salt-forming radicals.

9. An anti-static 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 a dialkyl phosphonic acid having the general formula:



wherein R' and R² are substituents selected from the group consisting of hydrogen and alkyl and R and R³ are substituents selected from the group consisting of alkyl, cycloalkyl, and heterocyclic groups and hydrogen, and Z is a substituent selected from the group consisting of hydrogen and salt-forming radicals.

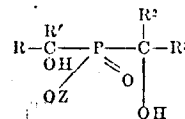
10. An anti-static composition adapted, when applied to cellulose organic derivative yarns, filaments, or fibers, to eliminate the tendency of such materials to accumulate charges of static

electricity, said composition containing as its essential anti-static component bis-beta-hydroxy propane-beta-phosphonic acid diethanolamine salt.

11. An anti-static 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 bis-beta-hydroxy butane-beta-phosphonic acid-diethyl-cyclohexylamine salt.

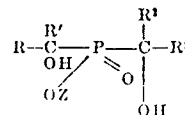
12. An anti-static composition adapted, when applied to cellulose organic derivative yarns, filaments or fibers, to eliminate the tendency of such materials to accumulate charges of static electricity, said composition containing as its essential anti-static component bis-beta-hydroxy pentane-beta-phosphonic acid ammonium salt.

13. Textile yarns having a lessened tendency to accumulate charges of static electricity, said yarns containing a salt of a dialkyl phosphonic acid having the general formula:



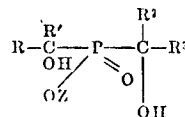
wherein R' and R² are substituents selected from the group consisting of hydrogen and alkyl and R and R³ are substituents selected from the group consisting of alkyl, cycloalkyl, and heterocyclic groups and hydrogen, and Z is a substituent selected from the group consisting of hydrogen and salt-forming radicals.

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 a dialkyl phosphonic acid having the general formula:



wherein R' and R² are substituents selected from the group consisting of hydrogen and alkyl and R and R³ are substituents selected from the group consisting of alkyl, cycloalkyl, and heterocyclic groups and hydrogen, and Z is a substituent selected from the group consisting of hydrogen and salt-forming radicals.

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 a dialkyl phosphonic acid having the general formula:



wherein R' and R² are substituents selected from the group consisting of hydrogen and alkyl and R and R³ are substituents selected from the group consisting of alkyl, cycloalkyl, and heterocyclic groups and hydrogen, and Z is a substituent selected from the group consisting of hydrogen and salt-forming radicals.

16. Textile yarns composed of or containing organic derivatives of cellulose having a lessened tendency to accumulate charges of static elec-

tricity, said yarns containing bis-beta-hydroxy propane-beta-phosphonic acid diethanolamine salt.

17. Textile yarns composed of or containing organic derivatives of cellulose having a lessened tendency to accumulate charges of static electricity, said yarns containing bis-beta-hydroxy butane-beta-phosphonic acid diethylcyclohexylamine salt.

18. Textile yarns composed of or containing organic derivatives of cellulose having a lessened tendency to accumulate charges of static electricity, said yarns containing bis-beta-hydroxy pentane-beta-phosphonic acid ammonium salt.

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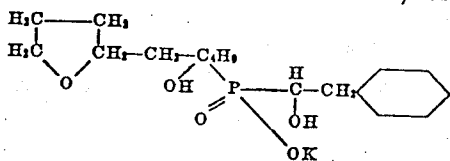
Certificate of Correction

Patent No. 2,279,502.

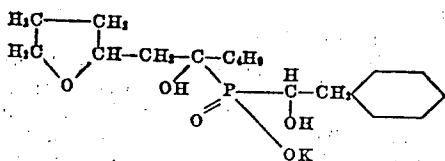
April 14, 1942.

JOSEPH B. DICKEY ET AL.

It is hereby certified that errors appear in the printed specification of the above numbered patent requiring correction as follows: Page 2, second column, line 2, for "99-100 parts" read *99-90 parts*; lines 66 to 71 inclusive, for



read



and that the said Letters Patent should be read with these corrections therein, that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 11th day of August, A. D. 1942.

[SEAL]

HENRY VAN ARSDALE,
Acting Commissioner of Patents.