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# HIGH TEMPERATURE RESISTANT TEXTILE FIBER FINISH COMPOSITION

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## ABSTRACT OF THE DISCLOSURE

A heat-stable finish composition for synthetic fibers which comprises

(1) a major portion of an alkali metal salt of a phosphate ester where the alcohol portion of the ester has from 6 to 10 carbon atoms, and

(2) a minor portion of a weak acid such as boric acid.

These finishes are applied to the fibers as dilute aqueous solutions.

The development of synthetic fibers having high heat-stability and the increased speed of processing have resulted in high temperature operations in order to give the fibers adequate temperature-time exposure. The temperatures of draw rolls, drying ovens and texturing gases has been increased to higher than 300° C. The upper limit of oxidative and thermal stability of conventional finishes has been reached and new materials or better stabilizers for old materials are needed.

Mineral oils and waxes; esters of fatty acids, dicarboxylic acids, or phosphoric acid, and certain alkylene oxide polymers and polyglycol ethers; have been used for lubricating both natural and synthetic fibers. Some of these also impart antistatic properties to the fibers. Finishes based on low molecular weight compounds are usually too volatile and are largely lost from the fiber and condense on walls of dryers and other cool surfaces and sometimes drip onto the fibers causing discoloration. Some of these finishes are solvents which remove paints and lacquers from the equipment. Finally, any of the thermally unstable finishes may smoke and fume obnoxiously. Satisfactory finishes should not hamper the carding and spinning of the fiber into yarns. Some finishes cause fibers to stick to rolls and cause roll wraps.

The present invention provides fiber finishes which are stable to the high temperatures required in high speed processing and which impart static resistance and lubrication to the fiber.

These results are achieved in the present invention by applying to synthetic fibers such as polyamides, polyesters or polyacrylics a finish made by combining an alkali metal salt of a phosphate ester with a non-volatile weak acid or salt of a weak acid. The esters of phosphoric acid are made from alcohols of 6 to 10 carbon atoms. A preferred ester is the dioctyl phosphate. This is used preferably in the form of its sodium or potassium salt.

The acid may be boric acid, polyacrylic, polymethacrylic or other weak acids such as polycrotonic acid, polyitaconic acid or long-chain fatty acids (e.g. about 16 to 20 carbon atoms) or salts of the fatty acids. The purpose of the



acid is to solubilize the alkali salt of the phosphoric ester.

The phosphate ester is generally present in amounts of about 75 to 90 parts by weight and the weak acid is generally present in amounts of about 25 to 10 parts by weight. The finish is applied to the fiber as an aqueous solution containing about one to twenty percent of the finish composition. The dry fibers generally have less than one percent by weight of the finish based on the fiber weight after the finish dries on the fiber. Commonly the finish on the fiber will run about 0.2 to 0.5 percent by weight.

The phosphate ester provides both lubrication and antistatic effects. If the chain length of the alkyl group is too long, the antistatic effect is poor. If the chain length is too short, then the lubricant action is poor. A dialkyl ester with alkyl chains of about 8 carbon atoms gives good antistatic protection as well as good lubrication. The alkyl groups need not be the same length and a mixture of $C_8$ and $C_{10}$ alcohols or $C_6$, $C_8$ and $C_{10}$ alcohols can be used.

The following test methods have been used for testing finishes for use in this invention. A sample of the finish is placed in the sample cell resting on the hot plate. A thermometer or a thermocouple is inserted in the sample cell.

In carrying out the test, the sample is heated at the rate of increase of 5° C. per minute. The temperature at which the sample first begins to smoke is recorded as the smoke point. The smoke density of 200° C. is estimated as light, medium, or heavy and recorded as the smoke density at 200° C.

Another test used to determine a good finish is the thin film oxidation-evaporation test which measures the tendency of a finish to build up on hot areas of the equipment. It is carried out by weighing out ~0.2 gram of the finish in an aluminum cup, heating for 16 hours at 200° C., determining the percent residue, and noting the appearance of the residue. A large hot plate (30.5 x 30.5 cm.) 12 x 12 inches, protected from drafts or a mechanical convection oven are the preferred means of heating. The residue may be negligible or massive, transparent and colorless or cloudy and dark. Many finishes leave an undesirable varnish-like residue.

Finishes suitable as antistats and lubricants, and stable at temperatures of 200° C. and higher are not common because if they are good antistats, they are usually of high molecular weight and thus leave heavy dark residues and if of low molecular weight, they are too volatile. Obviously, the chemical structure also affects the properties of finishes. A large number of materials tested failed in one requirement or the other. Some failed because the finish was a solvent for paints and lacquers.

It is therefore surprising that a finish was found that was satisfactorily free from all of these objections. The following examples will show the results of tests with finishes of this invention and also results with materials not meeting the requirements of this invention.

## EXAMPLE 1

A nylon yarn of 160 denier and having 68 filaments is spun from poly(hexamethylene adipamide) melt containing 0.5% $TiO_2$ and drawn to a 3.2 ratio. This yarn

is finished by passing it over a rotating roll which is partly immersed in a bath of the following composition:

| | Parts |
|---|---|
| Potassium salt of dioctyl phosphate | 16 |
| Boric acid | 4 |
| Water | 80 |

The yarn takes up about 30 parts of the finish for 100 parts yarn so that after drying, the yarn has 0.3% finish. The yarn is dried by passing through an oven at 250° C. and then bulked by passing through a steam jet as taught by U.S. Pat. 3,005,251 to Hallden. Bulking is accomplished with 1.9 lbs. ,0.86 kilogram) per hour of steam from a steam source at 60 pounds per square inch (4.2 kilograms per sq. cm.) at a temperature of 320° C.

No objectionable fuming or smoking occurs during the drying or bulking and no harmful deposits build up on the equipment. The yarn remains free of static after the finish is applied and is sufficiently lubricated to process with low running friction. When used as a filling yarn in weaving, it does not dissolve the varnish from the bobbins.

## EXAMPLE 2

A fiber is spun from a polymer of hydroxypivalic acid by the method of Example 4 of U.S. Patent 2,658,-055 of Alderson. The fiber in the form of continuous filament is treated with a solution of the following composition by passing the filaments over a rotating roll partly immersed in the solution:

| | Parts |
|---|---|
| Potassium salt of dioctyl phosphate | 0.75 |
| Potassium oleate | 0.25 |
| Water | 99.00 |

The fiber takes up 35 parts of this solution per 100 parts of fiber.

The fiber is next cut into staple and dried by passing through an oven with an air temperature of 300° C. No appreciable fuming occurs and no condensate forms on the cooler parts of the drier. The staple fiber is carded and spun into yarn with no difficulty from static and a low order of roll wraps on the drafting rolls.



## EXAMPLE 3

The following finishing compositions are prepared without water which would be used if the finishes were applied to fibers.

| | Parts |
|---|---|
| Finish 1: | |
| Potassium salt of dioctyl phosphate | 80 |
| Boric acid | 20 |
| Finish 2: | |
| Potassium salt of dioctyl phosphate | 90 |
| Polyacrylic acid (Acrysol A–1 manufactured by Rohm & Haas Co.) | 10 |
| Finish 3: | |
| Potassium salt of dioctyl phosphate | 80 |
| Potassium oleate | 20 |
| Finish 4: | |
| Potassium salt of dioctyl phosphate | 75 |
| Potassium oleate | 25 |

The smoke point, the smoke density at 200° C. and the thin film properties are determined by methods described above. The results are shown in Table 1 below. A conventional mineral oil finish is included for comparison.

TABLE 1

| | Finish 1 | Finish 2 | Finish 3 | Finish 4 | Conventional mineral oil finish |
|---|---|---|---|---|---|
| Smoke point ° C | 185 | 195 | 200 | 199 | 120 |
| Smoke density at 200° C | (1) | (1) | (1) | (1) | (2) |
| Thin film test, percent residue | 41 | 50 | 54 | 59 | 9.5 |
| Appearance of residue | (3) | (3) | (4) | (4) | (5) |

[1] Light.
[2] Heavy.
[3] Deliquescent friable light brown foam.
[4] Deliquescent friable brown granular solid.
[5] Dark brown varnish.

What is claimed is:

1. A high temperature resistant textile fiber finish composition which consists essentially of about 70 to 90 parts by weight of an alkali metal salt of a phosphate ester prepared from an alkyl alcohol having 6 to 10 carbon atoms and about 25 to 10 parts by weight of boric acid.


**References Cited**

UNITED STATES PATENTS

| | | | |
|---|---|---|---|
| 1,914,331 | 6/1933 | Nuesslein et al. | 252—8.6X |
| 2,025,435 | 12/1935 | Bou Huys | 252—8.8X |
| 2,289,127 | 7/1942 | Koch | 252—8.8X |
| 3,341,451 | 9/1967 | Dziuba et al. | 252—8.6 |
| 3,377,181 | 4/1968 | Kamijo et al. | 252—8.6 |



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