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3,704,225

**NONSWELLING TEXTURING SPIN FINISH**

Barry Michael Shay, Wilmington, Del., assignor to ICI America Inc.

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4 Claims

**ABSTRACT OF THE DISCLOSURE**

High-temperature stable alkyl stearate lubricant compositions which are especially useful as spin finishes in producing textured polyester yarns are obtained by forming aqueous emulsion blends with antistatic agents and thermal stabilizing agents using selective emulsifiers such as polyoxyethylene derivatives of sorbitol and sorbitan polyoleates. The finishes are superior in that they do not attack or swell polyurethane belts and cots used in commercially available false twist texturing equipment.

This invention relates to the finishing of textile fibers, filaments, and yarns, and more particularly to an improved spin finish composition for polyester yarns undergoing false twist texturing. In the production of synthetic continuous filament polyester yarns, it is necessary to apply to the filaments during the spinning operation a lubricating composition to reduce friction developed as the yarns pass over the metal and ceramic machinery surfaces. Since they are applied during the spinning step, they are frequently referred to as spin finishes. Such finishes usually contain a lubricating ingredient to reduce friction, an antistatic agent to reduce the build-up of static electrical charges on the surfaces of the yarn as it passes over various machinery components, an antioxidants or thermal stabilizing agent to reduce the build-up of resin deposits on the machinery and the yarn itself, and an emulsifying agent in order that the total system may be applied to the yarn from an aqueous emulsion.

Polyester yarns are best drawn and textured at elevated temperatures approaching 230° C. This higher temperature led to the problem of thermal degradation of the finish and the formation of impervious, difficult to remove varnish-like deposits on the equipment. While many formulations were devised to overcome the thermal instability of spin finishes, an additional problem was left unsolved. This problem relates not directly to the spin finish itself but rather to the material incorporated in the high-temperature false twist apparatus. This material, a polyurethane derivative, is employed in the construction of belts and cots used to grip and control the yarn as it passes through the texturing apparatus. Many prior high-temperature spin formulations are absorbed by the polyurethane belt material and cause their softening and swelling. As a result, the fiber wears a groove into the soft material and slippage results. When such slippage occurs, the apparatus must be removed from service and production stopped long enough to change the polyurethane parts.

Another improvement offered by the compositions of the invention is that they can be used not only as a filament spin finish but also as a combined spin and staple fiber finish in the production of polyester staple fiber. Usually spin finishes which are suitable for the drawing operation are not satisfactory as the second finish normally

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applied in producing polyester staple therefore, the spin finish is removed from the tow prior to the application of the staple finish. It is now possible with the application of the compositions of the invention to eliminate the finish removal step since the compositions serve equally well for both steps and, therefore, reduce the cost of the operation. Furthermore, if after completion of the spinning and drawing operation more lubricant is required in the preparation of staple from the yarn, the added finish need not contain the stabilizing agent referred to hereinbefore since this is not a high-temperature operation. Compositions otherwise conforming to the formulations hereafter described having no thermal stabilizing agent are thus considered to be within the purview of the invention.

It is an object of this invention to provide a textile finish suitable for high- and low-temperature processing of synthetic continuous filament and staple polyester yarns.

It is another object of this invention to provide a textile spin finish which will not degrade or swell polyurethane belts.

These and other objects are accomplished in a spin finish which is prepared by blending 22–70% by weight of a lubricant such as mineral oil, vegetable oil, pelargonate esters, stearate esters, with 5 to 35% by weight of antistatic agents such as potassium hexylphosphate, isopropylamine dodecyl benzene sulfonate, or diethyl sulfate quaternary of 1-hydroxy ethyl-2-heptadecenyl imidazoline; and forming an aqueous emulsion with water of the above ingredients using from 15 to 55% by weight of an emulsifier more fully defined hereinafter. Resistance to degradation of such emulsions is improved by the addition of up to 3% by weight of a thermal stabilizing agent such as 4,4'-bis-dimethyl benzyl diphenyl amine, or diphenyl amine derivative of acetone having a melting point of 85 to 95° C. and formed by reacting diphenyl amine with acetone at low temperatures.

As lubricants in the above general formulation, low molecular weight stearate esters of monohydric alcohols having 4 to 16 carbon atoms such as butyl stearate and isocetyl stearate and pelargonates such as the tripelargonate ester of trimethylolethane and trimethylolpropane are preferred.

The emulsifiers in the above general formulation are polyoxyethylene(2)oleyl alcohol, sorbitan trioleate, polyoxyethylene(20)sorbitan trioleate, and partial oleic acid esters of ethylene oxide adducts of hexitols or hexitol-water mixtures containing up to 15% by weight of water, such adducts containing from 90 to 93% by weight of oxyethylene radical, and said partial esters containing from 0.5 to 0.9 oleate radicals per hydroxyl radical of said adduct.

Representative of such partial oleic acid esters are the oleates obtained by esterifying oleic acid with polyoxyethylene adducts of hexitol or hexitol/water mixtures as indicated in Table 1.

The preferred formulations comprising lubricants, antistatic agents, emulsifying agents and, in some cases, antioxidants are blended and stored in concentrated form. However, finish compositions when applied in the spin, staple or texturing operation are preferably emulsified with warm water and form stable emulsions containing up to 98% water, but usually from 80–98% water. They are applied to single strands of the fiber which are coated with the emulsion to give an undiluted residual of spin finish equal to 0.1–1% by weight of the yarn.

TABLE 1.—PARTIAL OLEATE ESTERS OF ETHYLENE OXIDE ADDUCTS

Code:	Composition of adduct		
	Hexitol	Percent ethylene oxide content	Composition of esterified adduct: oleate/hydroxyl
I.....	Sorbitol.....	90.0	0.67
II.....	do.....	93.0	0.83
III.....	Mannitol (15% H <sub>2</sub> O).....	92.0	0.75
IV.....	Sorbitol (10% H <sub>2</sub> O).....	91.6	0.55
V.....	Sorbitol (15% H <sub>2</sub> O).....	90.6	0.75
VI.....	do.....	90.6	0.88
VII.....	do.....	92.4	0.75

## EXAMPLE 1

A stable aqueous emulsion is formed using a finish made by blending 58 parts by weight butyl stearate, 13 parts potassium hexyl phosphate (45% aqueous solution), 5 parts isopropylamine dodecyl benzene sulfonate, 2 parts 4,4'-bis-dimethyl benzyl diphenyl amine in 30% active dioxane solution (Naugard-445)<sup>1</sup> and 22 parts of the Partial Ester VI of Table 1. The emulsified finish is applied to 250/50/0 polyester yarn at a 1% by weight (measured as undiluted finish) level using a finish applicator (described in U.S. Pat. No. 3,347,207).

Treated yarn samples are conditioned overnight at 65% R.H. and 70° F. prior to determining their frictional properties at room temperature using a friction tester (U.S. Pat. No. 3,366,299) having a 1 cm. diameter, stainless steel pin and a yarn contact angle of 180° at yarn speeds of 10 and 50 meters /min. The coefficient of hydrodynamic friction (*f*) is calculated from the belt formula

$$\frac{T_2}{T_1} = e^{\theta}$$

where (*T*<sub>1</sub>) is the input tension of 15 gms. maintained on the yarn in advance of the pin, (*T*<sub>2</sub>) is the output tension on the yarn measured on the strain gauge after passing the pin, *θ* is the angle of contact (in radians), and (*e*) is the Napierian logarithm base.

Antistatic properties imparted by the finish are determined by measuring electrical resistance of polyester taffeta fabric (Testfabrics Style #704). Fabric is conditioned overnight at 40% R.H. at 75° F. prior to measuring its resistivity properties on a Beckman Ultrahmeter. Material treated with this spin composition at 0.5 and 1.0% levels give log of resistivity readings of 9.0 and 8.5, respectively.

Thermal stability of the finish is measured by placing a 1 gram sample in an aluminum dish heated on a hot plate at 420° F. for a period of 24 hours and noting the weight lost by the finish and the water solubility of the residue. In this case, the finish loses 70% and leaves an amber colored liquid which is water-soluble.

Swelling of polyurethane belt material is characterized by placing into an oven at 70° C. for 64 hours a 1-inch square section of the polyurethane belt having 6 to 10 drops of finish applied to it. The square is thereafter measured to determine the volume increase. With this finish, the degree of swelling is slight, amounting to a 1% increase.

## EXAMPLE 2

A stable aqueous emulsion is formed using a finish made by blending 50 parts by weight of butyl stearate, 30 parts isopropylamine dodecyl benzene sulfonate, and 20 parts of the Partial Ester V of Table 1. The finish is tested as described in Example 1. In the antistatic test, the finish gives Log of resistivity readings on polyester taffeta fabric of 11.2 and 10.4 where treatment levels of 0.5 and 1% are used. No visual or measurable swelling of polyurethane belt material is obtained.

<sup>1</sup> Commercially available from Naugatuck Chemical Co.

## EXAMPLE 3

A stable aqueous emulsion is prepared from a spin formulation containing 49 parts by weight of butyl stearate, 5 parts potassium hexyl phosphate (45% aqueous solution), 1 part Naugard-445, and 35 parts of Partial Ester V of Table 1. The formulation when tested as described in Example 1 proves to be a superior antistat as indicated by resistivity measurements of 8.8 and 8.4 for treatment levels of 0.5 and 1% when applied to polyester taffeta fabric. When examined for thermal stability, it yields a water-soluble, amber liquid after a weight loss of 70%. No measurable or visual swelling of polyurethane belt material is obtained.

## EXAMPLE 4

A stable aqueous emulsion is prepared from a spin formulation containing 30 parts by weight trimethylol-ethane tripelargonate, 30 parts potassium hexyl phosphate (45% aqueous solution), 22 parts polyoxyethylene (2) oleyl alcohol, and 18 parts of the Partial Ester V of Table 1. This formulation is a good lubricant and useful as a combination spin and staple finish.

## EXAMPLE 5

A stable aqueous emulsion is prepared from a finish having 40 parts by weight trimethylol-ethane tripelargonate, 15 parts potassium hexyl phosphate (45% aqueous solution), 30 parts of the Partial Ester V of Table 1, and 15 parts polyoxyethylene (20)-sorbitan trioleate. This formulation is a good lubricant and useful as a combination spin and staple finish.

## EXAMPLE 6

A stable aqueous emulsion is prepared from a finish having 40 parts by weight trimethylol-ethane tripelargonate, 15 parts diethyl sulfate quarternary of 1-hydroxyethyl-2-heptadecenyl imidazoline, 30 parts of the Partial Ester V of Table 1, and 15 parts polyoxyethylene (20) sorbitan trioleate. This formula is a good lubricant and useful as a combination spin and staple finish.

## EXAMPLE 7

A stable aqueous emulsion is formed from a finish having 58 parts by weight butyl stearate, 13 parts potassium hexyl phosphate (45% aqueous solution), 5 parts isopropylamine dodecyl benzene sulfonate, 22 parts of the Partial Ester VI of Table 1, and 2 parts of 30% dioxane solution of (Aminox)<sup>1</sup> a diphenyl amine derivative of acetone having a melting point of 85–95° C. and formed by reacting diphenyl amine with acetone at low temperatures. The finish is tested according to the procedures of Example 1 for thermal stability and gives a water-soluble black residue upon a weight loss of 70%.

## EXAMPLE 8

A stable aqueous emulsion is formed from a finish having 49% butyl stearate, 35% Partial Ester VII of Table 1, 15% potassium hexyl phosphate (45% aqueous solution), 1% Naugard-445, and applied as in Example 1. Upon a weight loss of 75%, it yields a water-soluble amber liquid.

## EXAMPLE 9

A stable aqueous emulsion is formed from a finish composition having 30% butyl stearate, 49% Partial Ester V of Table 1, 20% potassium hexyl phosphate, and 1% Naugard-445 in 30% dioxane solution. When tested as in Example 1, the finish yielded a water-soluble amber colored liquid upon a weight loss of 65%.

## EXAMPLE 10

A stable aqueous emulsion is formed from a finish composition having 69% butyl stearate, 25% of Partial Ester V of Table 1, 1% of a 30% dioxane solution of Naugard-445 and 5% isopropylamine dodecyl benzene sulfonate.

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EXAMPLE 11

A stable aqueous emulsion is formed of a finish having 49% isocetyl stearate, 35% of the Partial Ester V of Table 1, 10% sorbitan trioleate, 1% Aminox in 30% dioxane solution, and 5% isopropylamine dodecyl benzene sulfonate.

TABLE 2

	Yarn coating, percent by wt.	Yarn speed, m/min.	Coefficient of friction (f)	Yarn speed, m/min.	Coefficient of friction (f)
Example:					
1.....	1.0	10	0.43	50	0.68
2.....	1.0	10	0.66	50	0.91
3.....	1.0	10	0.51	50	0.73
4.....	0.15	10	0.61	-----	-----
5.....	0.15	10	0.73	-----	-----
6.....	0.5	10	0.76	-----	-----
7.....	1.0	10	0.43	50	0.68

What is claimed is:

1. A water emulsifiable textile finish which does not attack the polyurethane belts and cots used in false twist texturing equipment which consists essentially of:

(a) 22-70% by weight of a lubricant selected from the group consisting of butyl stearate and tripelargonate ester of trimethylolethane;

(b) 5-35% by weight potassium hexyl phosphate; and

(c) 15-55% by weight of an emulsifying agent selected from the group consisting of partial oleic acid esters of ethylene oxide adducts of sorbitol or sorbitol-water mixtures containing up to 15% by weight water, said adducts containing from 90-93% by weight of oxyethylene radical and said ester containing from 0.5-0.9 oleate radical per hydroxyl radical in said adduct.

2. A composition of claim 1 which consists essentially of 30% by weight trimethylolethane tripelargonate; 13% by weight potassium hexyl phosphate; 18% by weight of the partial oleic acid ester of an ethylene oxide adduct of an aqueous sorbitol solution containing 15% water, said

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adduct containing 93% by weight of oxyethylene radical, said ester containing 0.75 oleate radicals per hydroxyl radical of said adduct; 22% by weight of an additional emulsifier which is polyoxyethylene (2) oleyl alcohol; and 17% by weight water.

3. A composition of claim 1 consisting essentially of 45-65% by weight of butyl stearate lubricant; 6-35% by weight of potassium hexyl phosphate; 18-45% by weight of an emulsifier selected from the group consisting of (a) the partial oleic acid ester of an ethylene oxide adduct of an aqueous sorbitol solution containing 15% water, said adduct containing 91% by weight of oxyethylene radical, said ester containing 0.88 oleate radical per hydroxyl radical of said adduct; and (b) the partial oleic acid ester of an ethylene oxide adduct of an aqueous sorbitol solution containing 15% water, said adduct containing 93% by weight of oxyethylene radical, said ester containing 0.75 oleate radicals per hydroxyl radical of said adduct, and up to 15% by weight of water.

4. An aqueous emulsion of the composition of claim 1.

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HERBERT B. GUYNN, Primary Examiner

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