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3,201,362

TEXTILE FINISHES

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This invention relates to new compositions of matter which have exceptional properties when used as a textile finish, as a finish for thin, self-supporting films or as a coating on solid substrates. The invention also relates to the preparation of said new compositions.

More specifically this invention deals with a new composition of matter consisting of three components, one of which is a high molecular weight polymer whereas the two others are compounds of lower molecular weight, all three of which are brought together in such a manner that firm and permanent junctions are established between the individual molecules of the three components.

Still more specifically the invention is concerned with intimate blends of synthetic polymers as polyethylene, polypropylene, polystyrene, polyvinylchloride, polybutadiene-styrene or polybutadiene-acrylonitrile, with a textile plasticizer and a blend of a wax which is essentially hydrocarbonic in nature, comprising such substances as straight and branched aliphatic compounds or alicyclic systems, and contains a certain degree of polarity which is provided by the presence of such elements as oxygen, nitrogen, chlorine or fluorine, and a tacky elastomer.

It is well known that textile finishes consist essentially of three components: a plasticizer which penetrates the individual filaments of a yarn or fabric and renders them softer and more pliable; and adhesive, which binds the individual filaments together to form a firm and coherent yarn; and a lubricant which covers the surface of the yarns or cords, permitting them to run smoothly and rapidly over the various metallic guides of the textile machineries and protecting them against damage from friction and attrition. If the plasticizer is left out or does not function appropriately, the finished threads are harsh and wiry and do not come off smoothly from the shuttle. If the adhesive fails to make its full contribution, the yarns are fibrillated during weaving and, if the lubricant is not effective, the yarns don't run or stop readily and easily, but block and stick with the disastrous result that unnecessary heat is developed and very uneven and spotty fabrics are produced, even if one operates at slow speeds. Thus, each of the three components makes its important and necessary contribution to the functioning of the finish, the proper functioning of which evidently represents a very delicate balance between the individual contributions.

In general, the three components are a plasticizer, an adhesive and a lubricant, all three of which are dissolved or dispersed in water, or other vehicle, which carries them onto the fiber and which in some cases, such as with wool, cotton, rayon or polyvinylalcohol yarns, plays the role of a swelling agent or even as a mild and provisional plasticizer. However, simple dispersion or dissolution of the three essential ingredients of a textile finish in water or a substantially aqueous system such as water-methanol, water-alcohol, or water-acetone does not bring these materials in the sufficiently intimate and permanent relationship which is needed to guarantee their simultaneous deposition, firm, continuous adhesion and synergistic action during such prolonged and stringent operations as throwing, twisting, spooling, beaming, warping, weaving and knitting. In many instances, the individual components of the prior art finishes separate from each other during application on the goods and do not cover them uniformly and homogeneously. It must be remembered

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that all filamentous materials and polymers have a very large internal surface and, as a consequence exhibit a very strong separating capacity which is well known as the general phenomenon of chromatography and, particularly, as the still more striking and surprising effect of paper chromatography where one of the most important fiber forming polymers, namely cellulose, displays an unexpected capacity to separate organic compounds from each other by selective adsorption even if they are so similar to each other that they cannot be separated by any other method. Thus, the individual components of a finish are always exposed to the strong separating action of the filamentous material on which they are applied.

It is one important object of this invention to provide textile finishes wherein each of its components is chosen and blended with the others in such a manner that the chromatographic action of the textile material onto which the finish is applied separates the components in the most desirable manner, namely, in such a way that the *plasticizer component* penetrates rapidly and thoroughly into the interior of each individual filament, whereas the *adhesive component* spreads itself onto the surface of the individual filaments of the yarn and covers them with a thin and uniform layer and the *lubricating compound* finally is of such a character that it does not penetrate the structure of the yarn or cord, but remains concentrated and accumulated on the outer surface thereof where it provides a high degree of stickiness and lubricity.

Another object of this invention is the provision of textile finishes wherein each individual component is mutually compatible in the dissolved or dispersed state. Since these components have to be of different molecular size and different polar character, it is difficult to keep them together for the long periods during which textile finishes have to be stored especially under rather harsh conditions of extreme temperature changes, and exposure to vibrations and the action of its chemical ingredients. Whether in pure water or in a soap solution or in mixtures of water with organic liquids such as methanol, alcohol, acetone or others, the finishes of this invention remain homogeneous and immediately applicable even after long periods of storage under various conditions.

Still another embodiment of this invention is the provision of textile finishes which are advantageously employed on a wide variety of types of fibers and wherein the individual ingredients exhibit their characteristic activities not only for one special fiber or for a special type of fiber, such as the hydrophilic materials—wool, cellulose and polyvinylalcohol fibers—but also for a variety of fibers even if they belong to different characteristic groups such as the hydrophilic fibers, mentioned above, on one side and the hydrophobic fibers, such as nylon, Orlon, Dacron, Dynel, Creslan and Perlon, on the other side. This combination of ingredients, which serves both hydrophilic and hydrophobic fibers equally well, makes my finishes applicable to a variety of such fibers and also to blends of them either in the staple or in the yarn.

In order to set forth the characteristic features of this invention I shall first describe the choice of the three principal ingredients of the novel composition of matter and then disclose the special way in which these ingredients have to be combined with each other in order to arrive at the advantageous and superior performance of these new finishes.

The plasticizing component is a low molecular weight organic substance of the ester-, ether-, ketone or amide type, such as dioctyl phthalate, diethyleneglycol dimethyl-ether, diisopropylketone or diethylacetamide, which, in general, is a colorless liquid at around room temperature and can be dissolved or dispersed in water. The molecular weight of this component is between 50 and 1000 with

a preferred range between 100 and 300. Its boiling point is between 150 and 400° C. with a preferred range between 200 and 250° C. and its viscosity at room temperature is between 50 and 1000 centipoises with a preferred range between 100 and 500 centipoises.

The adhesive component consists essentially of a low molecular weight wax which contains a polar element such as O, N, F, or Cl; it is prepared by treating an aliphatic, cycloaliphatic or aliphatoaromatic hydrocarbon of low molecular weight with oxygen, nitrogen compounds, fluorine or chlorine until the desired degree of polarity is reached or the adhesive component consists essentially of a microcrystalline wax, e.g., Mekon*, or a polymerized microcrystalline, e.g., Polmekon* (see U.S. Patent 2,798,841). Typical polar waxes are the oxidized paraffinic waxes and the oxidized polyolefin waxes including the oxidized microcrystalline residues or the co-oxidized microcrystalline wax—polymerized unsaturated hydrocarbon mixtures until fully described in U.S. Patents Nos. 2,424,671; 2,573,422; 2,573,423 and 2,601,109; the oxidized polyolefins, e.g., oxidized polyethylene, the oxidized mixtures of microcrystalline wax and polyolefins such as those set forth in United States Patents Nos. 2,879,237 through 2,879,241; the oxidized Fischer-Tropsch waxes and the like. Examples of commercial oxidized waxes are the Cardis* waxes such as Cardis* No. 8, Cardis* No. 314, and Cardis* 935, Crown No. 23 and the like. The oxidized waxes are preferred. Also included in the polar waxes are the chlorinated waxes such as chlorinated paraffin waxes, the chlorinated naphthalenes, anthracenes, biphenyls, phenanthrenes, carbazoles and dibenzopyrones. In addition, the fluorinated waxes are useful in my novel compositions. Examples of fluorinated waxes are fluorinated paraffin, olefins, terpenes, naphthalenes, tetralins, anthracenes, biphenyls, phenanthrenes and carbazoles. Waxes containing nitrogen can also be employed. Such nitrogen-containing waxes are nitrated, nitrosated and aminated paraffins, olefins, naphthalenes, anthracenes and carbazoles. The molecular weight of this component is between 50 and 1000 with a preferred range between 100 and 300. It can be a waxy solid at room temperature with a melting point between 50 and 150° C. or a liquid at room temperature with a boiling point above 200° C. To this wax is added a small amount of a tacky elastomer such as rubber, Vistanex (polyisobutylene), Gentac (a copolymer of butadiene, styrene and vinylpyridine), Hevea rubber (cis-polyisoprene) and/or Estane (polyurethane rubber). The average molecular weight of this tackifying additive is preferably above 50,000 and the amount of it corresponds to 1 to 50 percent of the adhesive component with a preferred range between 2 and 8 percent. The two parts of the adhesive component must be intimately blended with each other before they are united with the other two essential ingredients of my novel finishes. This can be done by dissolving the wax and elastomer in a common solvent, usually an aliphatic or aromatic hydrocarbon, a chlorinated hydrocarbon, ester or ketone, and coprecipitating them by the addition of a non-solvent, preferably methanol, ethyl alcohol or acetone, or by emulsifying them jointly in water and then coprecipitating them by the addition of an acid—preferably sulfuric, hydrochloric or acetic acid. The coprecipitate represents either a soft tacky mass or a viscous tacky liquid with pronounced pressure sensitive adhesive characteristics. It can be added to the finish in form of a solution in one of the above-mentioned solvents or in another liquid which exhibits sufficient dissolution power, or in form of an emulsion, which can readily be prepared by stirring the material into an agitated soap solution.

The third, lubricating component of the finish consists of a vinyl polymer or copolymer which has film forming properties and represents, at, and slightly above, room temperature a tough, hard, smooth material which adheres tenaciously to the surface of textile goods. Pre-

ferred polymers are polyethylene, polypropylene, polyvinylchloride, polyvinylacetate, polymers of acrylic esters, polymers of methacrylic esters and acrylonitrile as well as the copolymers of the monomers, from which these polymers are made, with each other or other polymerizable unsaturated compounds. These polymers or copolymers can be used as they are. They can also, before use, be modified by oxidation, chlorination or hydrolysis until they become readily compatible with the two other, aforementioned, components of the finish. The average molecular weight of these polymers is high, at least 5000, the preferred range being between 10,000 and 30,000. The softening point is above 60° C., with a preferred range from 110 to 160° C. The lubricating component, which is responsible for the formation of a smooth protective film on the surface of the yarns or cords, can be incorporated into the finish in form of a solution or an emulsion in the same way as the other two components.

The term "polymer" as used herein is in its broad sense to include copolymers as well as polymers made from a single monomer. The molecular weights as set forth herein were determined cryoscopically or osmotically in cyclohexane or toluene.

The plasticizing component, adhesive component and the lubricating component as described above are blended in the form of a dispersion in a suitable liquid vehicle, i.e., as a solution in a solvent or as an emulsion. Water is the preferred liquid phase for the solution or emulsion, although mixtures of water with alcohols and other organic solvents, e.g., acetone, as well as other organic liquids or mixtures, can be advantageously employed. If a solvent or a mixture of solvents is employed, they must be chosen in such a manner that all three components of the finish are readily soluble in them and that the fibers, yarns and fabrics are not attacked by the solvent. Many combinations of solvents can be used. Preferred, are mixtures of hydrocarbons, chlorinated hydrocarbons, ketones, esters and/or ethers. If the deposition of the finish is to be carried out from an emulsion, the three components are coemulsified in a soap solution; if necessary with the addition of an additional surfactant and/or an emulsion stabilizer.

The proportions of the three components can be varied over a wide range but it has been found in the course of many tests that there exists for each fiber type a preferred range in which the most favorable effects are obtained. In Table I, where the results of a systematic investigation of such finishes are summarized, the relative amounts of the plasticizing component P, the adhesive component A and the lubricating component L are given in percent by weight of the aggregate weight of the three components. The last column of the table contains the preferred range for the total amount of finish applied in percent of the weight of the air dry fiber.

TABLE 1.—PREFERRED RANGE OF THE THREE COMPONENTS OF THE NEW FINISH FOR SEVERAL FIBER TYPES

Fiber type	Percentual range for component			Total amount of finish in percent of fiber
	P	A	L	
Celluloses.....	30-40	10-30	40-50	1-5
Acetate Rayon.....	20-30	10-30	50-60	2-5
Polyamides.....	30-40	20-50	20-40	1-3
Polyesters.....	30-40	20-40	30-40	1-3
Acrylics.....	30-40	20-40	30-40	2-4
Modacrylics.....	20-30	10-30	50-60	2-5
Vinylchloride.....	30-40	20-50	20-40	1-4
Polyethylene.....	10-20	20-40	50-60	2-4
Polypropylene.....	10-20	20-40	50-60	2-5
Polyvinylalcohol.....	30-40	10-30	40-50	2-5

If the above-mentioned three basic ingredients are merely blended together mechanically in a normal mixer by stirring, shaking or ball milling in the state of a viscous dope, a suspension or emulsion, one does not arrive

at a sufficient degree of uniformity which is needed to spread the resulting finish in a homogeneous manner uniformly over the surfaces of the fibers to which it is applied. The components of those systems are so incompatible and so well selected for synergistic action that they need a more intimate mixing, or homogenization, to be brought together to a sufficient degree.

As a result of systematic trials, I have found that it is best to carry out the mixing homogenizing operation with a colloid mill, an intensive blender or a high speed rotating blade mixer under such conditions that the total solid content of the final emulsion or suspension is between 30 and 70 percent with a preferred range between 50 and 60 percent. Certain emulsifiers or compatibilizers can be used to accelerate and facilitate the intimate mixing. Their quantity amounts to 1 to 10 percent of the solid content of the blend with a preferred range between 2 and 3 percent. They are the aliphatic esters, ethers or ketones with 4 to 22 carbon atoms in the chain with a preferred range between 12 and 18, e.g. dilauryladipate, dioctylsebacate, laurylpalmitate and the like.

The following examples illustrate some of the compositions which have been prepared in somewhat large quantities and have been intensively tested as finishes in various textile operations but they do not represent any limit for the formulation of textile finishes which are subject to this invention and which have been characterized and described in the text of this disclosure.

Example 1

Fifteen grams of the ammonium salt of monoctylsebacate are placed in a 2 liter flask in the form of a fine white powder. To it are added 42 grams of a fine powder of linear polyethylene having an average molecular weight between 12,000 and 15,000. The two powders are mixed intimately at room temperature by shaking the flask for 5-10 minutes on a shaking machine which rocks with 2-6 cycles per second. After this procedure 60 ml. of a 1.5 percent Ivory soap solution in water at 65° C. are added to the powder and a stirrer which rotates at about 600 r.p.m. is put into action in the flask. In another flask 18 grams of Mekon* Wax (a microcrystalline wax) are emulsified with 2.5 grams of tacky Hevea rubber in 30 ml. of water with the aid of 0.4 to 0.5 g. of Ivory soap at 65° C. with stirring or shaking over a period from 5-10 minutes and coprecipitated by the addition of HCl. The precipitated mass is re-emulsified as above and the resulting emulsion is added to the first flask. The resulting system which contains 15 grams of plasticizer, 18 grams of adhesive, 42 grams of lubricant and 1 to 2 grams of soap in 90 ml. of water is vigorously agitated in a colloid mill for 25 minutes at 75° C.

There results a creamy, stable emulsion which neither sets nor precipitates over a period of many weeks when kept in a closed container. If applied to textile goods, it produces a soft and pleasant yarn, superior cohesion of the individual filaments of the yarn and excellent lubricity of the yarns in the course of such textile operations as throwing, twisting, warping, weaving and so on.

Example 2

In a 2 liter flask are placed 25 grams of a fine powder of polyoxyethyleneglycol having a molecular weight between 500 and 700 and which has been half esterified with lactic acid. To it are added 95 grams of isotactic polypropylene which has been prepared with the aid of a $TiCl_3$, $AlEt_3$ catalyst in such a manner that its molecular weight ranges between 10,000 and 13,000. Polypropylenes of this type have been repeatedly described in the literature and they are obtained in the form of very fine, white powders. The two powders are intimately mixed with each other by shaking at room temperature for a period of 4-5 minutes. Then, there are added 230 ml. of water at 70° C. in which there are dissolved 1.6 grams

of ammonium oleate. The resulting system is agitated with an intensive blender for 18-20 minutes at a temperature of 75° C. whereupon it is transformed into a creamy emulsion. In another flask there is prepared an emulsion of 19 grams of Polmekon* (a polymerized microcrystalline wax) and 3.0 grams of Gentac in 45 ml. of a 0.8 percent Ivory soap solution by shaking or stirring the ingredients together for 10-15 minutes at 75° C. to form an emulsion which is then co-precipitated by the addition of a small amount of H_2SO_4 . The co-precipitated mass is then re-emulsified as described above, and this emulsion is added to the first system (in the 2 liter flask). The resulting mixture is vigorously agitated in an intensive blender for another 8-10 minutes at 60° C. The result is a creamy, very stable emulsion of about 50 percent solids solvent which is an excellent finish for textile goods made of nylon, Orlon, Dacron, Dynel or Acrilan.

Example 3

In a 2 liter flask are placed 25 grams of a fine powder of polyoxyethylene glycol having a molecular weight between 500 and 700 and which has been half esterified with lactic acid. To it are added 95 grams of isotactic polypropylene which has been prepared with the aid of a $TiCl_3$, $AlEt_3$ catalyst in such a manner that its molecular weight ranges between 10,000 and 13,000. Polypropylenes of this type have been repeatedly described in the literature and they are obtained in the form of very fine, white powders. The two powders are intimately mixed with each other by shaking at room temperature for a period of 4-5 minutes. Then, there are added 230 ml. of water at 70° C. in which there are dissolved 1.6 grams of ammonium oleate. The resulting system is agitated with an intensive blender for 18-20 minutes at a temperature of 75° C. whereupon it is transformed into a creamy emulsion. In another flask there is prepared an emulsion of 19 grams of Cardis* No. 8 (an oxidized mixture of petroleum wax and polyethylene) and 3.0 grams of Gentac in 45 ml. of a 0.8 percent Ivory soap solution by shaking or stirring the ingredients together for 10-15 minutes at 75° C. to form an emulsion which is then co-precipitated by the addition of a small amount of H_2SO_4 . The co-precipitated mass is then re-emulsified as described above and this emulsion is added to the first system (in the 2 liter flask). The resulting mixture is stirred in an intensive blender for another 8-10 minutes at 60° C. The result is a creamy, thick, very stable emulsion of about 50 percent solids solvent which is an excellent finish for textile goods made of nylon, Orlon, Dacron, Dynel or Acrilan.

In the above examples (1) Mekon* is a hard, brittle micro-crystalline wax having a melting point (A.S.T.M. D-127-30) of 190 to 195; penetration (100 g. 77°/5 sec.) of 3-5; color (NPA) of yellow to brown-black; an acid number of 0 and a saponification value of 0; (2) Polmekon* is microcrystalline wax treated according to United States Patent 2,798,841 and has a melting point (A.S.T.M. D-127-30) of 195 minimum; a penetration (100 g./77°/5 sec.) of 0-3; color (NPA) of yellow; acid number of 0 and saponification value of 0; and (3) Cardis* No. 8 is the product of co-oxidation of a mixture of a petroleum wax and polyethylene and has a melting point (A.S.T.M. D-127-30) of 205 to 210; penetration (100 g./77°/5 sec.) of 1-2; color (NPA) of 3-5; acid number of 7-10 and a saponification value of 24-28. It is made according to the teachings of U.S. Patent 2,471,102.

* Registered U.S. Patent Office by The Western Petrochemical Corp.

What is claimed is:

1. A textile finishing composition comprising a homogenized mixture of liquid dispersions of the ammonium salt of monoctylsebacate; polyethylene having a molecular weight between 12,000 and 15,000; and an

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intimate blend of a major amount of a microcrystalline wax and a minor amount of tacky poly(cis-isoprene).

2. Textiles treated with the composition of claim 1.

3. A textile finishing composition comprising a homogenized mixture of liquid dispersions of an ester of polyoxyethylene glycol having an average molecular weight between about 500 and about 700 and lactic acid; isotactic polypropylene having a molecular weight in the range of about 10,000 and about 13,000; and an intimate blend of a major amount of a potassium hydroxide treated, air-blown microcrystalline wax and a minor amount of tacky butadiene-styrene-vinylpyridine polymer.

4. Textiles treated with the composition of claim 3.

5. A textile finishing composition comprising a homogenized mixture of liquid dispersions of an ester of polyoxyethylene glycol having an average molecular weight of about 500 and about 700 and lactic acid; isotactic polypropylene having a molecular weight in the range of

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about 10,000 to 13,000; and an intimate blend of a major amount of a co-oxidized mixture of a microcrystalline wax and polyethylene and a minor amount of tacky butadiene-styrene-vinylpyridine copolymer.

6. Textiles treated with the composition of claim 5.

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