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3,694,257 POLYESTER COMPOSITIONS AND THEIR USE AS TEXTILE ASSISTANTS Thomas P. Dumont, Cincinnati, Ohio, assignor to Emery Industries, Inc., Cincinnati, Ohio No Drawing. Filed July 20, 1970, Ser. No. 56,726 Int. Cl. C10m 7/26 U.S. Cl. 117-139.5 F 9 Claims

ABSTRACT OF THE DISCLOSURE

A textile assistant and method of using same which comprises applying to a textile fabric an aqueous emulsion containing as the textile assistant, a polyester prepared from the reaction of polyols such as glycols with a dibasic or tribasic acid having from about 6 to about 54 carbon atoms, preferably a dimerized fatty acid. When tribasic acids are used, a glycol should be employed and when triols are used, dibasic acids should be employed.

BACKGROUND OF THE INVENTION

This invention is concerned with polyester textile assistants and a method for their use, particularly as soften- 25 ing agents.

Within the past decade considerable progress has been made in the textile finishing art wherein textile assistants are applied to various textile materials derived from both natural and synthetic fibers to render the fibers more amenable to textile operations or to provide a product which will better serve the user. An ideal textile assistant beneficially modifies the properties of the textile fibers and is not removed or altered by ordinary washing or drycleaning procedures. The assistant preferably should be liquid at room temperature and readily dispersible in aqueous media used in textile processing or finishing, or otherwise readily applicable to fibers or textiles. Once applied, the assistant should have the property of retention on the fiber without harmful effect such as discolora- 40 tion, odor, or decreased light fastness of dyed fabrics.

Among the many types of textile assistants may be mentioned softeners used in both regular (non-resin) and resin finishing of textile to impart a soft full hand and to provide fabrics with enhanced drape. The desired properties in such softeners include stability against heat, oxidation and odor (rancidity) development, resistance to yellowing or scorching, freedom from deleterious effect of light upon the fastness of dyes, and the right lubricity to maintain or improve fabric sewability. Softeners of anionic, cationic, and nonionic types have been developed which provide a number of these desirable properties, but most are deficient in one respect or another. For example, the anionic sulfonated tallows and oils are subject to odor development through rancidity. These anionics and also 55nonionics generally have less permanency on the fiber than cationics. However, the cationics for the most part have a deleterious effect on the light fastness of dyestuffs. Furthermore, many of these types lack stability against either heat or light under textile processing, storage, or 60 use conditions and these drawbacks have hampered their use

Polyester textile assistants of this invention can be made liquid at room temperature which helps to facilitate their use. They possess excellent surface active properties, are 65 readily water dispersible for general use as emulsifiers, wetting agents, detergents, or dispersing agents. Also they are suitable as textile softeners, lubricants, and otherwise as textile assistants in dyeing, washing, scouring, stripping, and finishing.

When used as softeners, the textile assistants of this invention demonstrate a high degree of stability, resistance

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to yellowing and scorching which is frequently diminshed in the softening treatment. They also impart good sewability, lubricity, tensile and tear strength properties to the textile materials.

DESCRIPTION OF THE INVENTION

The present invention provides a method whereby textile materials are made more amenable to textile operations or are given certain desired properties which en-10 hance their value, such as softness to touch. The improved properties of the textile materials are achieved by the treatment of the textile materials with low molecular weight polyesters formed from the condensation of certain polybasic acids and polyols.

The polybasic acids which may be used in the present invention include aliphatic polybasic acids having from about 6 to 54 carbon atoms, specific examples of which are azelaic acid, dimer acid, and trimer acid. The preferred polyesters for the purposes of this invention are prepared using dimer acid. Dimer and trimer acids are polymerized 20 unsaturated fatty acids having about 36 and 54 carbon atoms and 2 and 3 carboxyl groups respectively. They may be formed by the polymerization of such unsaturated fatty acids as oleic acid. linoleic acid, linolenic acid, and the like, according to well known polymerization methods, such as are described in U.S. Pats. Nos. 2,482,761 2,793,-219, and 2,955,121. Dimer acid is usually sold commercially as a mixture of dimer and trimer acids, the trimer content ranging up to about 25 percent by weight of the total acid. The polybasic acids may be used either alone 30 or in mixtures with other polybasic aliphatic acids.

The polyols used in the formation of the polyesters of this invention are lower aliphatic polyhydroxy alcohols, such as ethylene glycol, propylene glycol, 1,4-butanediol, 35 neopentyl glycol, glycerol, polyproplene glycol, and polyehtylene glycol (molecular weight about 90 to 600). The preferred polyol is ethylene glycol and thus the preferred polyester for purposes of this invention is one prepared from ethylene glycol and a dimer acid. Ethylene glycol enables the preparation of polyesters having excellent textile fabric modifying properties and is relatively inexpensive.

The polyesters of the present invention may be obtained by heating the reactants in the presence of a conventional esterification catalyst, such as phosphoric acid, hypophosphorus, sulfuric acid, or paratoluene sulfonic acid.

When the polybasic acid is reacted with a polyoxyalkylene glycol, such as polyoxyethylene glycol, or polyoxy- $_{50}$ propylene glycol, it is preferable that the glycol possess an average molecular weight of from about 90 to about 120. Polyoxyalkylene glycols having a molecular weight up to about 600 may also be employed, but those falling within the 90 to 120 range are preferred as softeners.

The polyesters of the present invention should preferably have an average molecular weight of from about 1000 to 3000; however, polyesters with molecular weights above 3000 may be used up to the point which they become too viscous to enable their effective use.

Preferably the average hydroxyl functionality per molecule of the polyols and the average carboxyl functionality of the polybasic acids should be about two. When reactants of higher functionality are used, the polybasic acid and polyol components used in the preparation of the polyesters of the present invention should be selected in such a manner that a dibasic acid or one having an average functionality of about 2.3 or less is used when a polyol or polyol mixture having an average hydroxyl functionality greater than 2.3 per molecule is used, and when a polybasic acid having a functionality greater than about 2.3 per molecule, e.g. trimer acid which has a functionality of about 3 per molecule, is used, a glycol or mixture of polyols having an average functionality of about 2.3 or less should be employed. The proper selection of polybasic acids and polyols is necessary to avoid any high degree of cross-linking which would result in the gelation of the polyester reaction product and render the composition unuseable for the purposes of the present invention. The polyesters should be substantially linear in structure in order that they will have a low viscosity and be effective for the purposes of the invention.

As has been previously indicated, the polyesters of the present invention are advantageously utilized as textile assistants, particularly as textile softeners which when applied to fabrics impart to the fabric excellent scorch resistance, increased tensile and tear strength, and improved sewability characteristics. Certain of these polyesters may also be employed in other applications which require a nonionic surface active agent or agents of high thermal stability, for example, wetting and rewetting agents in textile processing.

20Textile materials which may be treated with textile assistants in accordance with the present invention include fibers, yarns, and fabrics of natural and synthetic origin. Materials may be cellulosic in nature, such as cotton, rayon, or acetate, or blends of the same. Synthetic textile 25materials, such as nylons, acrylics, polyesters, polyurethane (Spandex), polyolefins, and mixtures of those materials with natural fibers may also be beneficially treated in accordance with the invention. Softeners of the invention are particularly applicable in regular and resin finishing of cotton and synthetic textiles or blends. The specific polyesters of the present invention may be used in a variety of forms depending upon their intended function. They may be used in the form of an aqueous emulsion, or they may be used in the form of an organic 35 solvent solution.

In general, when the polyesters are intended for use as textile softeners, it is preferable to prepare them in the form of aqueous dispersions, and they may be used in this form in regular finishing, resin finishing, top 40 softening and back filling. The aqueous dispersions are prepared using a nonionic emulsifier, such as polyoxyalkylated alkyl phenols, e.g. nonyl phenol ethoxylated with 9 to 10 mols of ethylene oxide if the particular polyester requires it. In addition to the aqueous dispersion comprising water, the emulsifier, and the polyesters 45 of this invention, other additives may be added which are commonly used in the textile industry, such as resins, catalysts, wetting agents, and builders. The softening compositions do not require the use of additives such as oils or other lubricants which reduce viscosity 50 or provide fiber to metal lubrication. The softening compositions of the invention provide fiber to fiber lubricity as distinguished from fiber to metal lubricity.

The specific technique chosen to apply the textile assistants to textile materials varies with its function. 55 A preferred technique for softeners involves padding to impregnate the material with a dilute aqueous dispersion of the assistant. After the softener composition has been applied, it is bonded to the fabric. In regular finishing the bonding is of a physical nature and is accomplished by 60 drying at temperatures of about room temperature to about 200° C.

In resin finishing the textile fabric, after application of the softening composition by such means as padding is heated at a temperature of about 20 to 200° C. in the 65 presence of an aminoplast resin to effect a chemical bonding of the softener to the resin and fabric. Typical concentrations for a dilute aqueous solution of the polyester assistant in a softener application range from about 0.5% to 4% assistant by weight. With resin finishing 70 for crease-proofing fabrics, curing temperatures from about $150-200^{\circ}$ C. for a period of about 2 to 15 minutes are recommended for best results. Textiles treated in accordance with the invention exhibit resistance to discoloration under heat, improved hand or touch prop-75

erties and tear strengths and improved sewability characteristics, when compared to samples using textile assistants without a softening agent.

Where used as lubricants, the assistants may be applied in numerous ways, e.g. by spraying, wicking, or dipping as commonly used and well known in the art. The polyesters when used primarily as textile lubricants are generally applied from aqueous dispersions.

The invention and some of its advantages are further illustrated in the following examples which provide complete, specific embodiments of the invention, but which are not intended to be limitations thereof.

EXAMPLE I

A mixture of 83 grams of ethylene glycol, 507 grams of dimerized fatty acid sold under the trademark Empol 1018, and having a dimer acid content of about 83% and a trimer acid content of about 17%, and 0.1% H₃PO₂—50% catalyst were slowly heated to a maximum of 240° C. with agitation. The water of esterification was drawn off and the mixture was held under these conditions until an acid value below 5 and a hydroxyl value of between 50 to 60 was obtained. The resulting polyester product was in the form of a viscous oil.

75 grams of the polyester product prepared in the method described above was then blended with 25 grams of an ethoxylated nonyl phenol emulsifier. $\frac{1}{2}$ gram of the resulting mixture was then dissolved in 99.5 grams of water and padded onto 80 picks x 80 picks cotton fabric, rayon velvet fabric, and $\frac{65}{35}$ polyester/cotton fabric, at room temperature. All of the treated fabrics when dried, possessed a soft, silky touch to the hand and had no significant loss of scorch resistance.

EXAMPLE II

A mixture of 385 grams of polyoxyethylene glycol (molecular weight 600) and 240 grams of a dimerized fatty acid sold under the trademark Empol 1018, and 0.1% H_3PO_2 —50% catalyst were slowly heated to a maximum of 260° C. with agitation. The water of esterification was drawn off and the mixture was held under these conditions until an acid value of 5.3 was obtained. $\frac{1}{2}$ gram of this polyester was then dissolved in 99.5 grams of water and padded onto an 80 picks x 80 picks cotton fabric at room temperature. The treated fabric, when dried, showed improved touch to the hand and had no significant loss of scorch resistance, but the improvement was less than noticed with the composition used in Example I.

EXAMPLE III

A mixture of 90 grams of ethylene glycol, 190 grams of azelaic acid, and 0.1% of a H_3PO_2 -50% catalyst were slowly heated to a maximum of 240° C. with agitation. The water of esterification was drawn off and the mixture was held under these conditions until an acid value below 5 and a hydroxyl value of 56 was obtained. A hard, white, waxy product was obtained. 75 grams of the resulting polyester product was then blended with 25 grams of ethoxylated nonyl phenol emulsifier. 1/2 gram of the resulting mixture was then dissolved in 99.5 grams of water and padded onto 80 picks x 80 picks cotton fabric at room temperature. The treated fabric, when dried, had a pleasing touch to the hand and had no significant loss of scorch resistance, but the improvement was slightly less than the improvement over the untreated material which was realized by the treatment of the fabric with the materials and in the manner described in Example I.

EXAMPLE IV

0.5% to 4% assistant by weight. With resin finishing 70 for crease-proofing fabrics, curing temperatures from about 150–200° C. for a period of about 2 to 15 minutes are recommended for best results. Textiles treated in accordance with the invention exhibit resistance to discoloration under heat, improved hand or touch prop-75 tion. The water of esterification was drawn off and the set of the text of text of the text of the text of text of text of the text of t

mixture was held under these conditions until an acid value of 5.2 was obtained. The resulting polyester was a viscous brown liquid. 75 grams of the polyester were blended with 25 grams of a nonionic emulsifier. 1/2 gram of this solution was then dissolved into 99.5 grams of 5 water and padded onto 80 picks x 80 picks cotton fabric at room temperature. The treated fabric, when dried, possessed a soft and silky touch to the hand and showed no significant loss of scorch resistance.

EXAMPLE V

A mixture of 105 grams of 1,4-butanediol, 472 grams of a commercially available dimerized fatty acid sold under the trademark Empol 1018 and 0.1% by weight of reactants of H₃PO₂-50% catalyst were slowly heated to 15 a maximum temperature of 255° C. with agitation. The water of esterification was drawn off and the mixture was held under these conditions until an acid value of 2.5 and a hydroxyl value of 38 were obtained. The resulting polyester was a viscous, yellow oil. 75 grams of this polyester 20 fatty acid is dimer acid. were then blended with 25 grams of the nonionic emulsifier used in Example I. 1/2 gram of this mixture was then dispersed in 99.5 grams of water and padded onto 80 picks x 80 picks cotton fabric at room temperature. The treated fabric, when dried, possessed a pleasant touch to 25 the hand and had no significant loss of scorch resistance.

EXAMPLE VI

A mixture of 720 grams of commercially available triand 580 grams of diethylene glycol were heated in the presence of 0.1% by weight of H3PO2-50% catalyst based on the weight of the reactants were heated to a maximum of 240° C. with agitation. The water of esterification was drawn off and the mixture was held under 35 these conditions until an acid value of 5.6 and a hydroxyl value of 118 were obtained. 1/2 gram of the resulting polyester product was dissolved in 99.5 grams of isopropanol-91% and padded onto an 80 picks x 80 picks cotton fabric at room temperature. The treated fabric, after drying, had 40 a pleasant, soft, silky touch to the hand and showed no significant loss in scorch resistance.

Obviously, many modifications and variations of the invention as hereinbefore set forth may be made without departing from the spirit and scope thereof, and there- 45 fore, only those limitations should be imposed as are indicated in the appended claims.

I claim:

1. A method for softening textile fabrics which comprises applying to said fabric a polyester obtained by the 50 T. G. DAVIS, Assistant Examiner condensation of a polymerized fatty acid having from about 36 to 54 carbon atoms and a polyol or mixture of polyols, the polymerized fatty acids and polyol components being selected in such a manner that polymerized fatty acids having an average functionality of about 2.3 55

per molecule or less are used when said polyol has a functionality greater than about 2.3 per molecule and a polyol or mixture of polyols having an average functionality of about 2.3 per molecule or less is used when said polymerized fatty acids have a functionality greater than about 2.3 per molecule, and then bonding said polyester to said fabric.

2. The method of claim 1 wherein said bonding is performed by heating said fabric with said polyester 10 applied thereto in the presence of an aminoplast resin at a temperature of about 20° C. to 200° C. to effect a chemical bonding of said polyester to said resin and to said fabric.

3. The method of claim 1 wherein said polyol is a glycol selected from the group consisting of polyethylene glycol, ethylene glycol, propylene glycol, neopentyl glycol, 1,4-butanediol, polypropylene glycol, and mixtures thereof.

4. The method of claim 1 wherein said polymerized

5. The method of claim 1 wherein said polymerized fatty acid is trimer acid.

6. The method of claim 1 wherein said polymerized fatty acid is a mixture of dimer and trimer acids.

7. The method of claim 1 wherein said polymerized fatty acid is dimer acid and said polyol is ethylene glycol. 8. The method of claim 1 wherein said polyester has a molecular weight from about 1,000 to about 3,000.

9. A softened textile fabric comprising a textile fabric merized fatty acid sold under the trademark Empol 1040 30 having bonded thereto a softener composition consisting essentially of a polyester obtained by the condensation of a polybasic acid or mixture of said acids having from about 6 to 54 carbon atoms and a polyol or mixture of said polyols, the polybasic acids and polyol components being selected in such a manner that polybasic acids having an average functionality of about 2.3 per molecule or less are used when said polyol has a functionality greater than about 2.3 per molecule and a polyol or mixture of polyols having an average functionality of about 2.3 per molecule or less is used when said polybasic acids have a functionality greater than about 2.3 per molecule.

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WILLIAM D. MARTIN, Primary Examiner

U.S. Cl. X.R.

117—138.8 D, 138.8 E, 138.8 F, 138.8 N, 138.8 UA, 139.4, 143 A, 161 K; 252-8.6, 8.9

UNITED STATES PATENT OFFICE CERTIFICATE OF CORRECTION

Patent No.	3,694,257		Dated	September	26,	1972
Inventor(s)	Thomas P.	Dumont				

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

On the cover sheet insert -- This application is a continuation in part of Application S/N 657,081 filed July 31, 1967 entitled 'POLYESTER COMPOSITIONS AND THEIR USE AS TEXTILE ASSISTANTS' --.

Signed and sealed this 20th day of August 1974.

(SEAL) Attest:

McCOY M. GIBSON, JR. Attesting Officer C. MARSHALL DANN Commissioner of Patents

FORM PO-1050 (10-69)