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# FINISHES FOR TEXTILE FABRICS

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## ABSTRACT OF THE DISCLOSURE

Crease-resisting and soil-releasing finishes for textile fabrics comprise esters of polyalkylene glycols alone or combined with copolymer acid emulsions, said finishes being curable by irradiation.

This invention relates to new textile finishing compounds, to the process for treating a substrate to impart improved crease-resistance and soil-release properties thereto, and to the textile materials finished therewith.

In recent years rapid strides have been made in achieving fabrics with commercially valuable properties. Wash-and-wear materials that shed wrinkles and require only a light ironing were followed by durable-press fabrics which can be laundered repeatedly without losing their crease and without requiring ironing. The latter, however, have a marked deficiency; namely, they become soiled when they come into contact with hydrophobic materials such as paraffin oils, vegetable oils, dirty motor oil, food colors, and the like. This is particularly the case with polyester or polyester-blend fabrics where the inherent characteristics of the polyester plus the permanent press treatment result in a material that is hydrophobic and oleophilic. Not only does the fabric attract dirt and/or oily materials and retain them, but also the soil that has been removed from the fabric during the washing is redeposited onto the material as an even film, Furthermore, water, detergents, optical brighteners, and the like have difficulty penetrating the fibers of the fabric and getting it clean. As a result, after having been laundered a garment tends to gray or yellow due to the soil and/or oily materials that have been deposited and remain thereon; additional use and washing of the garment increase the intensity of the discoloration.

The textile industry consequently has conducted extensive research on anti-soiling. Numerous patents have issued covering the utilization as a soil-release agent of both inorganic and organic materials; the following being representative: the process of U.S. Pat. 2,734,835 (Florio et al.) employs hydrous stable metal oxides. U.S. Pat. 2,999,774 (Shappel) relates to the utilization of silica particles and a salt of a multivalent metal to render a fabric soil resistant. U.S. Pat. 3,089,778 (Pierce et al.) discloses utilizing a water-insoluble basic aluminum salt having an ultimate particle size of less than 0.5 micron. U.S. Pat. 3,152,920 (Caldwell et al.) claims the use of a composition comprising a polymeric polybasic acid cross-linked with the reaction product of an organic polyisocyanate and a polyol. U.S. Pat. 3,236,685 (Caldwell et al.) involves the application of a solution of a polymeric polybasic acid (—COOH, —$SO_3H$, or —$PO_4H_2$) and a polyol or a polyepoxide. U.S. Pat. 3,322,569 (Faulhaber et al.) teaches as the soil-release agent a copolymer of a N-methylolamide of an acrylic or methacrylic acid, a compound containing at least two polymerizable double bonds in the molecule, and an alkyl acrylate or methacrylate. U.S. Pat. 3,362,782 (Gagliardi) discloses treatment of the textile with a heterocyclic nitrogen base. U.S. Pat. 3,377,249 (Marco) relates to imparting soil-release and durable-press characteristics to a polyester-containing textile by means of an aminoplast textile resin, a textile resin catalyst, and a synthetic acid



emulsion polymer comprising at least 20 weight percent of an acrylic acid.

While the application of such soil-release finishes to durable-press fabrics does have some beneficial effect in resisting soil and in retaining whiteness, problems involving their use remain. For example, the activity of these and other known soil-release finishes diminishes after repeated washing; they do not satisfactorily inhibit soil redeposition in the laundering cycle; they are deleteriously affected by softeners; they lessen fabric strength and abrasion resistance; the use of such materials as aminoplast resins may be hazardous due to the fumes of the unreacted resin; fluorocarbons are relatively expensive and tend to impart a harsh hand to fabrics; and some finishing agents cause excessive dusting and deposition of the soil-release agent on sewing needles and other equipment.

It has now been found that these disadvantages may be overcome and that a crease-resistant and soil-releasing finish can be imparted to a wide variety of textile materials by treating them with esters of polyalkylene glycols. The resulting fabrics, which may be 100% cotton, rayon, wool, linen, paper, nylon, polyester, acrylic, polyamide, polyurethane, polyolefin, and the like, and blends thereof, are characterized by excellent soil-release, crease-resistance, and recovery from wrinkles without loss of strength and abrasion resistance.

The esters of this invention have the general formula $RnO(XO)_m$, where R is selected from the group consisting of acrylyl, methacrylyl, itaconyl, crotonyl, and the like, and mixtures thereof; X is an alkylene radical or mixture of alkylene radicals having 1 to 4, and preferably 2 to 3, carbon atoms per radical; *n* is an integer from 2 to 12, and preferably 2 to 6; and *m* is an integer from 1 to 10, and preferably 3 to 4. Suitable esters include, for example, the di-, tri-, and tetraacrylates; the di-, tri-, and tetramethacrylates; the di-, tri-, and tetraitaconates; the di-, tri-, and tetracrotonates; and the like of polyethylene glycol, polypropylene glycol, neopentyl glycol, hexanetriol, ethoxylated glycerine, ethoxylated sorbitol, the Pluronics (block copolymers of ethylene oxide and propylene oxide having varying amounts of the ethylene oxide and propylene oxide components, sold by Wyandotte Chemicals Corp.) and the like.

The polyalkylene glycol esters may be applied to the textile fabrics alone or in combination with any suitable copolymer acid emulsions. The copolymers may be prepared from any polymerizable organic acid and a monomer copolymerizable therewith. Examples of the polymerizable acids include acrylic acid, maleic acid, fumaric acid, methacrylic acid, itaconic acid, crotonic acid, cinnamic acid, polymerizable sulfonic acids, polymerizable phosphoric acids, etc. Monomers that may be interpolymerized with the acids include any monomers that are capable of copolymerizing with the acids and which will not detrimentally affect the film-forming properties of the polymer. Suitable monomers include esters of the above acids with an alkyl alcohol, e.g., ethyl acrylate, methyl acrylate, propyl acrylate, isopropyl acrylate, methyl methacrylate, ethyl methacrylate, 2-ethylhexyl acrylate, butyl acrylate, etc.; alkyl fumarates, maleates, crotonates, cinnamates, etc.; vinyl halides; monomers having vinylidene groups, e.g., styrene, acrylonitrile, methylstyrene; substituted vinyl monomers, e.g., chlorostyrene; butadiene; etc.; and mixtures thereof.

Some of the synthetic acid polymers that may be used according to the present invention are polymerization products of ethyl acrylate:acrylic acid; ethyl acrylate: acrylic acid:acrylamide; butyl acrylate:acrylic acid:ethyl acrylate:methacrylic acid; ethyl acrylate:itaconic acid;

methyl methacrylate:acrylic acid; 2-ethylhexyl acrylate: acrylic acid; butyl acrylate:acrylic acid:acrylamide; ethyl acrylate:acrylic acid:N-methylol acrylamide; ethyl acrylate:acrylic acid:styrene; ethyl acrylate:acrylic acid:hydroxypropyl methacrylate; ethyl acrylate:acrylic acid: divinyl benzene; ethyl acrylate:acrylic acid:allyl acrylamide; ethyl acrylate:acrylic acid:glycidyl acrylate; ethyl acrylate:itaconic acid; ethyl acrylate:sodium styrene sulfonate; ethyl acrylate:crotonic acid; styrene:acrylic acid; ethyl acrylate:acrylic acid:hydroxyethyl methacrylate; hydroxyethyl methacrylate:acrylic acid:acrylamide; butyl acrylate:ethyl acrylate:acrylic acid; and the like.

The amount of the copolymer acid emulsion employed may range from 0 to about 10 percent, based on the total weight of the textile, and is preferably about 0.5 to 5 percent. When the copolymer acid emulsion is used in combination with the ester, the ratio of ester to copolymer may vary from 0.5–10:1, and is preferably about 1–3:1.

Although the use of a catalyst is not required, one may be included if desired in order to activate the reaction between the ester and the substrate. Materials such as peroxides or azo compounds may be used in an amount ranging from about 0.001 to 5.0, and preferably about 0.1 to 0.5, percent, based on the weight of the ester. Examples of suitable catalysts include dilauroyl peroxide, t-butyl perpivalate, t-butyl perbenzoate, t-butyl peracetate, t-butyl peroxide, and other compounds of comparable free radical activity, and mixtures thereof.

The polyalkylene glycol esters of this invention are generally, but not necessarily, applied to the substrate as a solution in, for example, an alcohol, such as methyl alcohol, ethyl alcohol, isopropyl alcohol, and the like; a mixture of water and alcohol; or water.

The bath used to impregnate the fabric according to the present invention is not limited to the ingredients mentioned above, namely, the polyalkylene glycol ester in the presence or absence of a solvent, an acid polymer, and/or a catalyst. Other ingredients may be included, if desired, such as for example emulsifying agents, wetting agents, softeners, and so forth, that enhance the physical characteristics of the fabric.

In general the esters of this invention are applied to the fabric by padding from a solution containing from about 1 to about 10, and preferably about 5 to 7, percent by weight of the ester to give 100% wet pickup onto the fabric. It is to be understood that other settings of the padder to permit different concentrations of the ester in the padding bath are permissible in order to achieve the same net add-on of the active ingredients. On a dry basis, the weight of the ester applied generally corresponds to about 1 to about 10 percent of the weight of the fabric.

The finishing compound of this invention may be applied to any suitable substrate, for example, films, sheets, fibers, yarns, threads, fabrics (knitted, woven, or nonwoven), or a product made therefrom, e.g., a garment. It may be applied in any convenient manner, for example, by spraying, padding, dipping, or the like. This invention will, however, be described in relation to the preferred embodiment, i.e., padding onto a substrate in fabric form.

Garments made from fabrics treated by the process of this invention need no steps other than the conventional ones for the preparation of durable-press garments. The garment may be folded and pressed on conventional equipment, e.g., a Hoffman press. A standard pressing cycle is used, generally involving pressing the garment for a short time and then curing.

An important feature of this invention is that the curing is accomplished by irradiation. The process involves employing radiation having sufficient energy to remove an electron from a gas atom, forming an ion pair; this requires an energy of about 32 electron volts (e.v.) for each ion pair formed. This radiation has sufficient energy to non-selectively break chemical bonds; thus in round numbers radiation with energy of 50 electron volts (e.v.) and above is effective for the process of this invention, although energies of 50,000 e.v. and over are preferred. Both particle radiation and ionizing electromagnetic radiation are included.

The preferred radiation is high energy ionizing particle radiation; energy equivalent to at least 0.1 million electron volts (mev.) is preferred. Higher energies are even more effective; there is no known upper limit, except that imposed by available equipment.

The high energy particle radiation is an emission of highly accelerated electrons, neutrons, alpha particles, deuterons, beta particles, or the like directed so that the said particle impinges upon the polymer.

Similarly, ionizing electromagnetic radiation (X-rays) useful in the process of this invention is produced when a metal target, e.g., gold or tungsten, is bombarded by electrons possessing appropriate energy, e.g., 0.1 mev. In addition to X-rays produced as indicated above, ionizing electromagnetic radiation suitable for carrying out the process of the invention may be obtained from a nuclear reactor ("pile") or from natural or artificial radioactive material, for example, cobalt 60.

The dose rate ("intensity" of dose) is not critical, being primarily a matter of available equipment. In general, high dose rates are preferred as promoting higher throughput.

The preferred dosage of irradiation for the practice of the present invention is the range of about 1000 rads to 50 megarads and more preferably in the range of about 0.3 to 5 megarads.

Efficiency of dose utilization may be improved by keeping the substrate and the treating material in contact for an extended time after irradiation to provide maximum opportunity for the radical-initiated chains to grow.

The sequence of treating the fabric is not critical; the fabric may be padded, dried, and then irradiated; padded, irradiated, and then dried; padded and irradiated; or the like. For evaluation purposes the material was then washed in a typical home laundry washing machine and conditioned for at least 24 hours at about 73° F. and 50% R.H. before testing. The duration of the various processing steps will vary depending upon the ingredients employed. The treatment time must, however, be sufficiently long to cause the ester to become firmly bonded to the fabric. In general the drying and irradiating steps are conducted at a rate of about 0.5 to 10 feet per second and the drying temperature, when employed, is about 135° to 250° F.

In the following examples after each padded sample was irradiated it was laundered and conditioned and then tested for retention of the padded additive, tensile strength, wrinkle recovery angle, soil-release, and other properties as follows:

*Laundering* was done in one home washing in a Kenmore washer, using ¼ cup of All (a commercial detergent) at a water temperature of 140° F.

*Conditioning* was done as described in AATCC Method 66–1959T, ASTM Method D–1295–67, Federal Specification CCC–T–191B (method 5212), and American Standards Association Method L–22, except that the temperature was 73±1° F. and the relative humidity was 50±2%.

*Wrinkle Recovery* (WRA) was determined by AATCC Method 66–1959T, ASTM Method D–1295–67, Federal Specification CCC–T–191B (method 5212), and American Standards Association Method L–22.

*Soil Release* was determined by AATCC Method 130–1969.

*Strength* was determined by ASTM Method D–1682–64.

The more detailed practice of the invention is set forth in the following examples. These examples are illustrative only and are not intended to limit the invention except as indicated by the appended claims. Unless otherwise specified, all parts are given by weight.

## EXAMPLE 1

Samples of 80 x 80 cotton fabric were padded with a solution comprising 40 parts of polyethylene glycol (200) diacrylate and 360 parts of 32% aqueous methanol (10% diacrylate in 32% methanol), dried, and then subjected to irradiation by a beam of high energy electrons produced by a 300 kev. Dynacote particle accelerator (manufactured by Radiation Dynamics, Inc.) to a total dosage of 4 megarads. Next the samples were laundered, dried, and finally conditioned at 73° F. and 50% R.H. An overall gain in weight of 5.8% was noted. The wrinkle recovery angle was 232° and the tensile strength was 60 p.s.i. $(w+f)$.

The control, i.e., untreated laundered, conditioned fabric, showed 180° wrinkle recovery and 66 p.s.i. tensile values.

## EXAMPLE 2

The procedure of Example 1 was repeated except that the cotton fabric was padded with a variety of compositions, as summarized below:

TABLE I

| Pad bath composition | Dosage (megarads) | WRA (°) | Tensile (p.s.i.) |
|---|---|---|---|
| None | 4 | 180 | 66 |
| 10% tetraethylene glycol diacrylate in 32% methanol | 1.0 | 230 | 67 |
| 5% neopentyl glycol diacrylate in 100% methanol | 0.5 | 228 | 71 |
| Hexanetriol triacrylate in 100% methanol | 1.0 | 239 | 78 |
| Pluronic L-31 diacrylate in 100% methanol | 0.5 | 236 | 70 |

## EXAMPLE 3

The procedure of Example 1 was repeated except that the fabric was Dacron T-54 [1]/cotton (65/35) and the padding compositions varied, as summarized below:

TABLE II

| Pad bath composition | Dosage (megarads) | WRA (°) | Tensile (p.s.i.) |
|---|---|---|---|
| None | 2.0 | 266-276 | 67 |
| 10% tetraethylene glycol diacrylate (TEEGDA) in 32% methanol | 2.0 | 309-318 | 64-65 |
| 5% TEEGDA and 3% of a commercial acrylic copolymer emulsion (Rohm & Haas Co.'s Rhoplex SR-488) in 32% methanol | 2.0 | 298-301 | 81-84 |
| Ethoxylated glycerine triacrylate in 100% water | 0.5-1.0 | 293-316 | 69-78 |
| 5% and 10% triethylene glycol dimethacrylate in 100% methanol | 0.5-1.0 | 286-307 | 68-78 |
| Ethoxylated sorbitol hexacrylate in 100% water | 0.5-1.0 | 290-321 | 68-77 |
| 5% polyethylene glycol (200)* diacrylate in 32% methanol | 1.0 | 293-315 | 67-75 |
| 5% polyethylene glycol (200) diacrylate in 100% methanol | 1.0 | 293-315 | 67-75 |
| 10% polyethylene glycol (200) diacrylate in 32% methanol | 1.0 | 296-320 | 67-75 |
| 10% polyethylene glycol (200) diacrylate in 100% methanol | 1.0 | 296-320 | 67-75 |
| 5% TEEGDA in 32% methanol | 1.0-1.5 | 270-302 | 71-82 |
| 10% TEEGDA in 32% methanol | 1.0-1.5 | 277-289 | 71-76 |
| 5% polyethylene glycol (300)* diacrylate in 32% methanol | 1.25 | 308 | 75 |
| 10% polyethylene glycol (300) diacrylate in 32% methanol | 0.5 | 290 | 70 |
| 5% polyethylene glycol (400)* diacrylate in 32% methanol | 1.25 | 293 | 76 |
| 10% polyethylene glycol (400) diacrylate in 32% methanol | 1.25 | 292 | 69 |
| 5% and 10% neopentyl glycol diacrylate in methanol | 0.5-1.0 | 289-321 | 63-73 |
| 5% and 10% hexanetriol triacrylate in methanol | 0.5-1.0 | 288-311 | 63-76 |
| 5% and 10% Pluronic L-31 diacrylate in methanol | 0.5-1.0 | 291-306 | 66-73 |
| 5% and 10% Pluronic L-35 diacrylate in water | 0.5-1.0 | 288-320 | 66-77 |
| 5% and 10% Pluronic L-44 diacrylate in water | 0.5-1.0 | 283-320 | 65-80 |

* The diacrylate of polyethylene glycol having a molecular weight of about the amount in parentheses.

[1] Dacron T-54 is a polyester fiber manufactured by E. I. du Pont de Nemours & Co.

## EXAMPLE 4

The procedure of Example 1 was repeated except that the fabric was Kodel [2]/cotton (65/35) broadcloth and the padding composition varied, as summarized below:

TABLE III

| Pad bath composition | Dosage (megarads) | WRA (°) | Tensile (p.s.i.) |
|---|---|---|---|
| None | 2.0 | 270 | 78-81 |
| 5% and 10% tetraethylene glycol diacrylate (TEEGDA) in 32% methanol | 0.5-1.25 | 297-306 | 74-82 |
| 10% TEEGDA and 3% of an acrylic copolymer emulsion | 0.5-1.25 | 266-279 | 79-81 |

## EXAMPLE 5

The soil release properties of fabrics treated with the compounds of this invention were determined as follows:

Two samples were taken of each of the treated fabrics. The samples of one set were stained with mineral oil and then subjected to one home washing in a Kenmore automatic washer using one cup of All and a water temperature of 140° F. The samples of the second set were first washed five times under wash conditions identical to those of the first set; they were then stained with mineral oil and subsequently subjected to one additional wash under the same conditions.

After each wash the samples were dried in a Kenmore automatic dryer for about 30 minutes. The residual stains in the samples were compared to a set of standards having numerical ratings of 1.0 to 5.0, 1.0 being very poor and 5.0 being substantially complete removal of the stain. Ratings are tabulated below and indicate the soil release property of the treated fabrics. Samples designated A, B, and C were, respectively, 100% Dacron taffeta, 65/35 Dacron/cotton shirting, and 65/35 Kodel/cotton broadcloth.

TABLE IV

| Sample | Soil release rating after— wash | 5 washes |
|---|---|---|
| A—untreated | 2.5 | |
| A plus 5% tetraethylene glycol diacrylate (TEEGDA) | 3.5 | |
| A plus 5% TEEGDA and 3% of an acrylic copolymer emulsion | 4.5-5.0 | |
| B—untreated | 3.0 | 2.5 |
| B plus 5% TEEGDA | 4.5-5.0 | |
| B plus 10% TEEGDA | 3.0-3.5 | 4.5 |
| B plus 5% TEEGDA and 3% of an acrylic copolymer emulsion | 5.0 | 3.0 |
| B plus 7.5% TEEGDA and 3% of an acrylic copolymer emulsion | 5.0 | 4.5 |
| C—untreated | 3.5 | 3.5 |
| C plus 10% TEEGDA | 3.0-3.5 | 4.5-5.0 |
| C plus 5% TEEGDA and 3% of an acrylic copolymer emulsion | 5.0 | 3.0-4.0 |
| C plus 7.5% TEEGDA and 3% of an acrylic copolymer emulsion | 4.5-5.0 | 3.5-4.0 |

## EXAMPLE 6

The procedure of Example 5 was repeated except that each of the following was used instead of tetraethylene glycol diacrylate: neopentyl glycol diacrylate; hexanetriol triacrylate; ethoxylated glycerine triacrylate; ethoxylated sorbitol hexacrylate; and the diacrylates of Pluronics L-31, L-35, and L-44. The results were comparable.

As can be seen from the above table, in addition to having excellent wrinkle recovery properties, the compounds of this invention have excellent soil release properties. They are applicable to a variety of substrates and impart durable press and soil release properties to a substrate without loss of strength and abrasion resistance.

[2] Kodel is a polyester fiber manufactured by Eastman Kodak Co.

What is claimed is:

1. A process for imparting crease-resisting and soil-release characteristics to a textile substrate selected from

the group consisting of cellulosic textiles, polyesters, and mixtures thereof which comprises the steps of (1) applying thereto a composition comprising (a) a compound selected from the group consisting of tetraethylene glycol diacrylate, neopentyl glycol diacrylate, hexanetriol triacrylate, ethoxylated glycerine triacrylate, ethoxylated sorbitol hexacrylate, and polyethylene glycol diacrylate and (b) a copolymer acid emulsion prepared by polymerizing a monomeric mixture of a lower alkyl acrylate or a lower alkyl methacrylate and acrylic acid or methacrylic acid in the amount of about 1 to 10 percent based on the dry weight of the substrate and (2) exposing the thus-treated textile substrate to about 1000 rads to 50 megarads of high energy radiation equivalent to at least 0.1 million volt.

**2.** The process of claim **1** wherein the compound is applied as a solution.



**3.** A textile material having crease-resisting and soil-release characteristics prepared by the process of claim **1.**


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