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# METHOD OF TREATING FABRICS

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U.S. Cl. 8—115.5 9 Claims

## ABSTRACT OF THE DISCLOSURE

This invention concerns a process wherein fabrics are impregnated with a durable press type resin containing, in addition to the resin and catalyst, an inorganic salt and a poly glycollate compound.

This invention relates to methods of treating fabrics to provide a durable finish fabric having improved soil resist and soil release properties.

## BACKGROUND OF THE INVENTION

This invention pertains to fiber and fabric finishes, and particlarly to resin finishes which impart durable finish or press properties to the fabric. Wash and wear or durable press garments have become increasingly popular in the consumer market, particularly in the areas of men's shirts and trousers and women's sport wear. The most popular fabrics are those composed of polyester or cotton, and especially polyester-cotton blends. The durable press properties are imparted to the fabrics by impregnating the fabric with a resin compound, then curing the resin while the fabric is held in the desired shape, for example flat, creased, or pleated. Once cured, the resin is durable to washing and dry cleaning, and serves to return the fabric to the desired shape after laundering or other distortion has occurred.

The advantages of the durable press fabrics are clearly easy care and a neat, wrinkle-free appearance. The primary disadvantage of the resin treatment is the increased tendency for the fabric to pick up and hold soil, especially oily soil. This undesirable property of durable press fabrics is characterized by a general graying of the fabric over an extended period of use accompanied by the formation of heavy soil lines where the fabric contacts the body of the wearer, such as at the cuffs and collar of a man's shirt.

Efforts by the synthetic fiber and garment manufacturers to eliminate the soiling propensity of durable press fabrics have resulted in some improvement. In French Pat. 1,401,581 a process is described for treating polyester articles using a polymeric crystalline compound that is insoluble in water, the compound containing a first portion made up of repeating units identical with those forming the crystalline portion of the polyester and a second portion having at least one active group such as polyoxyethylene, the treament being effected by applying the compound to the surface of the polyester filaments and curing the combination at a temperature greater than 90° C. in French Pat. 1,455,242 the same crystalline compound is applied to the polyester at ambient temperature in the presence of a polyester swelling agent.

It is generally recognized that the polyglycol finishes, by imparting hydrophilic and oleophobic properties to hydrophobic fibers significantly improve the resistance to soiling of the treated fabrics. Under proper conditions, sodium poly glycollate may be reacted with polyethylene terephthalate in an ester interchange which results in the poly glycollate becoming chemically bonded to the surface of the fiber. The result is a durable finish which imparts the desired hydrophillic and oleophobic properties to the fiber. However, there is no commercial application of this method since the ester interchange reaction with poly glycollate is difficult to control.



A commercial method for applying polyglycol to the surface of polyester fibers consists of the treating the fabrics with an aqueous dispersion of a low molecular weight polyoxethylene terephthalate polymer prepared by reacting terephthalic acid or dimethyl terephthalate with ethylene glycol and polyethylene glycol. The polymer is incorporated in the aqueous resin solution for a one step application to the fabric. Generally, the fabric is padded with the resin-glycol mixture and dried and cured while held in the desired configuration.

Fabrics treated with the combination resin-glycol mixture have appreciably less tendency to soil or form heavy soil lines than the durable press fabrics treated with resin alone. However, even these improved fabrics are generally more prone to soiling than fabrics which have not been resin treated. In order to satisfy consumer demands, it is therefore desirable to produce a resin treated durable press fabric which has soiling resistance and soil release properties at least equal to and preferably superior to the untreated fabric.

## SUMMARY OF THE INVENTION

Polyester, cotton, and polyester/cotton blend durable press fabrics having excellent soil resistant and soil release properties are otbained by impregnating the fabric with an aqueous solution ocntaining a resin and catalyst, an inorganic salt, and a dispersion of a low molecular weight polyoxyethylene polymer. The resin is any one of several recognized durable press resins such as the modified ureas, urea formaldehyde, and melamine formaldehyde. The inorganic salt is preferably calcium chloride or potassium chloride. The polyoxyethylene polymer dispersion is produced by reacting dimethyl terephthalate with ethylene glycol and polyethylene glycol.

It is therefore an object of this invention to produce a resin treated durable press fabric having superior soil resist and soil release properties.

It is a further object of this invention to produce a resin treated durable press fabric which is hydrophillic and oleophobic in nature.

Another object of this invention is to produce a durable press polyester/cotton resin treated fabric which is resistant to graying caused by gradual soil deposition.

Yet another object of this invention is to provide a composition of matter which may be applied to a fabric composed of polyester and/or cotton fibers to render the fabric both durable press and soil resistance.

A further object of this invention is to provide a novel and improved method of treating durable finish fabrics to improve the soil and stain release properties of the fabrics.

Still another object of this invention is to provide a method of treating a durable finish fabric to prevent graying of the fabric caused by the gradual accumulation of soil on the fabric.

These and other objects of this invention will become apparent from the following detailed description of the invention.

## DESCRIPTION OF THE PREFERRED EMBODIMENTS

In the practice of this invention, the fabric to be treated is impregnated with the resin solution and dried and cured in accordance with standard textile finishing procedures. The fabric may be impregnated by padding, spraying, or any other method, although padding is generally preferred for convenience of application. In the

padding procedure, the squeeze roll pressure is preferably set so that the wet pick up is about 50% of the dry weight of the fabric. The impregnated fabric may be dried and cured in a hot air oven by pressing on a hot head press with equally effective results.

The finish solution may contain from 5 to 25 percent resin (on a solids basis), the preferred amount depending upon the type and contemplated end use of the fabric being treated. For relatively light weight fabrics normally used in the manufacture of men's shirts for example, 5 percent resin may prove sufficient for the desired properties, although generally 10 percent is prefered. On the other hand, when treating the heavier fabric destined for use in trousers and the like, 25 percent or more resin may be required in the finish solution to produce adequate durable press properties. In practice, the levels of resin required for various fabrics and garments are well established in the industry, and the normal levels may continue to be used in the practice of this invention.

The durable finish resin used may be any of the modified ureas, urea formaldehyde, or melamine formaldehyde resins commonly employed for durable press effects. A partial list of some of the more popular resins includes dimethylol dihydroxy ethylene urea, dimethylol dihydroxy propylene urea, cyclic ethylene urea-formaldehyde, glyoxal urea-formaldehyde, dimethylol urea, dimethylol ethylene urea, and other similar resins known to be effective in the treatment of fabrics.

The resin is applied to the fabric in the presence of a catalyst to promote the cross linking and curing of the resin. Suitable catalysts well known in the trade include, among others, magnesium chloride and nitrate, and zinc chloride and nitrate. The amount of catalyst used is generally 5 to 15 percent by weight of the resin. Fabrics are generally dried at about 190–210° F. to leave 5–8% moisture. The fabrics are then cured at 250–400° F. and preferably 275–340° F. for 1.5–15 minutes depending on fabric weight, color, and style.

The low molecular weight polyoxyethylene terephthalate polymer, hereinafter referred to as POET, to be used in conjunction with the resin and inorganic salt, may be produced by reacting terephthalic acid or dimethyl terephthalate with ethylene glycol and polyethylene glycol. One procedure for preparing such compounds is described in Example 1 of French Pat. 1,455,242 as follows:

194 parts of DMT, 155 parts of EC, 440 parts of PEG 1540, 0.44 part of 2,6-ditertiarybutyl-1-methyl phenol, 0.1512 part of calcium acetate, and 0.0388 part of antimony trioxide are heated between 194° C. and 234° C. over a period of 4½ hours during which 57 parts of methanol are distilled off. Phosphorous acid (0.141 part of a 24.8% solution in EG) are added, and the molten material is heated at 282° C. in a polymerization tube. After removal of excess glycol in a rapid nitrogen stream, the pressure is reduced to 0.1 mm. Hg and the polymerization is continued for 15 minutes. The resulting product (relative viscosity 1.33, measured as before) softens at 40 to 50° C. and melts at 199 to 200° C.

The ratios of reactants are not critical, and may be varied over wide limits to vary polymer solubility and molecular weight. The following procedure illustrates the flexibility which is permissible in the preparation of the POET polymer:

194 parts of DMT, 500 parts EG, and 0.250 part of zinc acetate were heated to 190° C. with stirring while under nitrogen and maintained at this temperature until no more methanol was distilled. The pressure was gradually lowered to 1 mm. Hg while excess glycol was distilled off. 600 parts of molten PEG-1000 was then added and distillation continued up to a maximum of 165° C. The resulting product was a white, waxy solid.

The inorganic salt applied to the fabric simultaneously with the resin and POET polymer in accordance with



the method of this invention is one selected from the group consisting of chlorides, nitrates, and sulfates of metals found in Groups I–A and II–A of the Periodic Table of Elements. Of particular importance are calcium chloride and potassium chloride, which have been found to produce very good results with not apparent undesirable secondary effects. The nitrates, for example, have the disadvantage of causing the fabrics to discolor during the curing process. Certain salts of magnesium and zinc, primarily the chlorides and nitrates act as catalysts upon the resin and decrease the useful life of the resin solution when the combined finish is applied from a single bath.

It has been found that good results are obtained when the concentration of salt in the padding bath is between 5 and 15 percent, and preferably about 10 percent, at concentrations below 5 percent, full benefit of the invention in producing soil resistant fabrics is not realized. Concentrations greater than 15 percent may be used but little if any advantage is gained by operating at these higher concentrations.

In addition to the resin, catalyst, POET polymer, and inorganic salt ingredients specified above, the padding bath may also contain other textile finishing agents such as polyethylene based softeners. These are incorporated primarily to modify the fabric hand or body, and are not critical to the practice of this invention.

In a preferred embodiment of the practice of this invention, the fabric to be treated is padded with an aqueous solution of the selected resin, catalyst, POET polymer, inorganic salt, and optionally other textile finishing agents. After being passed through squeeze rolls to remove the excess liquor, the fabric is dried and cured to set the resin.

The soil resist and soil release properties of the fabric are determined by measuring the original whiteness of the treated fabric, soiling the fabric under standard laboratory conditions and remeasuring the whiteness, and finally washing the fabric and measuring the washed whiteness. The terated fabrics are scoured prior to measuring the original whiteness in order to remove the inorganic salt originally applied with the resin.

The whiteness of the fabric samples are measured on the Hunterlab Model D–40 Reflectometer for Whiteness, manufactured by the Hunter Associates Laboratory Inc., of McLean, Va. Whiteness values are calculated according to the procedure set forth by the manufacturer.

The fabric samples are soiled using a standard laboratory soiling emulsion and method. The soiling emulsion is prepared by melting together the following ingredients:

| | Percent |
|---|---|
| Palmitic acid | 10.0 |
| Stearic acid | 5.0 |
| Coconut oil | 15.0 |
| Paraffin | 10.0 |
| Spermaceti | 15.0 |
| Olive oil | 20.0 |
| Squalene | 5.0 |
| Cholesterol | 5.0 |
| Oleic acid | 10.0 |
| Linoleic acid | 5.0 |

To this melted mixture is added:

| | Grams |
|---|---|
| Oleic acid | 4.0 |
| Triethanalamine | 8.0 |
| Airborne soil | 4.0 |

The components are mixed thoroughly and added to an equal volume of 130° F. water in a Waring Blendor set at about 9,000 r.p.m. The mixture is homogenized for 15 minutes, then is diluted to a liter and homogenized 15 minutes longer.

The fabrics are then soiled by vigorously shaking the fabric and soiling emulsion together in a container with a ratio of soiling emulsion to fabric of 100 to 1. After 5

minutes agitation, the fabric is removed from the emulsion and passed through squeeze rolls to remove excess liquor, allowing a wet pickup of about 75–80% of the weight of the dry fabric. The fabrics are finally oven dried for 10 minutes at 185° F. and the soiled whiteness measured on the D–40 instrument.

Soiled fabrics are washed at 120° F. for 10 minutes using a liquor ratio of 20/1 and 1/0 grams/liter Tide detergent. The fabrics are rinsed in warm water and tumble dried prior to determining the washed whiteness values.

The practice of this invention is further illustrated in the following examples, where all parts and percentages are by weight unless otherwise specified. The examples are intended to illustrate the invention, and the invention is not to be considered limited thereto.

## EXAMPLE I

Samples of oxford cloth composed of 65 percent polyester and 35 percent cotton were padded with various aqueous solutions of a durable press resin, dried, cured, and evaluated for original whiteness and for soiling and soil release characteristics. The resin solutions evaluated were as follows.

Solution 1:
10% dimethylol dihydroxy ethylene urea
1.1% zinc nitrate catalyst
4% polyethylene base textile softener
Solution 2:
Solution 1 plus 20% POET
Solution 3:
Solution 1 plus 10% POET and 10% $CaCl_2$

After padding, the weight of the wet fabrics was approximately 50 percent greater than the weight of the dry fabric. The amount of resin and other finish ingredients applied to the fabric may therefore be easily calculated if desired. The wet fabrics were subsequently oven dried for 5 minutes at 200° F. and then cured for 10 minutes at 325° F. The treated fabrics were finally scoured in a standard solution of 0.1 percent Tide detergent, rinsed in warm water, and dried for 10 minutes at 200° F.

The whiteness of the treated fabrics was measured using the Hunterlab D–40 Reflectometer, after which the samples were soiled using the Spangler Procedure as set out above. The soiled samples were measured for whiteness, then washed, dried, and remeasured for whiteness according to the procedure set out above. The original whiteness, soiled whiteness, and washed whiteness for each of the resin solutions are presented in the following table. Data for an untreated control sample is included for purposes of comparison.

TABLE I

| Solution | Fabric whiteness | | |
|---|---|---|---|
| | Original | Soiled | Washed |
| 1 | 109 | 65 | 73 |
| 2 | 103 | 66 | 86 |
| 3 | 92 | 70 | 98 |
| Control | 112 | 66 | 84 |

It is apparent from the above data that the fabric treated with Solution 3 which contained both the POET and the $CaCl_2$ had significantly better soiled and washed whiteness values than any of the other three samples. In other words, the fabric had less tendency to soil and also had considerably better soil release properties than the comparison samples.

## EXAMPLE II

The identical procedure of Example I was repeated with fabric swatches composed of 100 percent cotton fibers in place of the polyester/cotton blend fabric. The original whiteness and the soiling and soil release properties of the treated fabrics are compared with an untreated control in the following table:

TABLE II

| Solution | Fabric whiteness | | |
|---|---|---|---|
| | Original | Soiled | Washed |
| 1 | 113 | 68 | 76 |
| 2 | 109 | 68 | 76 |
| 3 | 105 | 67 | 91 |
| Control | 124 | 77 | 113 |

As in Example I, the fabric treated with Solution 3 containing both the POET and the $CaCl_2$ salt had a soiled whiteness value significantly better than that obtained with Solutions 1 and 2. The difference between the original whiteness and the soiled whiteness shows Sample 3 had the least tendency to soil and the difference between the soiled and washed whiteness values shows Sample 3 had the best soil release of any of the treated samples.

## EXAMPLE III

Swatches of oxford cloth composed of 65 percent polyester and 35 percent cotton fibers were treated with various resin solutions, dried and cured, scoured and redried, and finally evaluated for soiling and soil release as described in Example I. The resin solutions evaluated were as follows.

Solution 1:
10% dimethylol dihydroxy ethylene urea
1.1% zinc nitrate catalyst
Solution 2:
Solution 1 plus 20% POET
Solution 3:
Solution 1 plus 10% POET and 10% $CaCl_2$
Solution 4:
10% cyclic urea-glycol resin
1.0% magnesium chloride catalyst
Solution 5:
Solution 4 plus 20% POET
Solution 6:
Solution 4 plus 10% POET and 10% $CaCl_2$
Solution 7:
10% dimethylol dihydroxy propylene urea
1.5% zinc nitrate catalyst
Solution 8:
Solution 7 plus 10% POET and 10% $CaCl_2$
Solution 9:
10% dimethylol urea
1.5% zinc nitrate catalyst
Solution 10:
Solution 9 plus 10% POET and 10% $CaCl_2$

TABLE III

| Solution | Fabric whiteness | | |
|---|---|---|---|
| | Original | Soiled | Washed |
| 1 | 106 | 64 | 78 |
| 2 | 103 | 67 | 90 |
| 3 | 112 | 71 | 102 |
| 4 | 114 | 71 | 91 |
| 5 | 111 | 71 | 96 |
| 6 | 114 | 72 | 103 |
| 7 | 97 | 59 | 67 |
| 8 | 113 | 66 | 90 |
| 9 | 99 | 60 | 69 |
| 10 | 116 | 65 | 89 |
| Control (avg.) | 114 | 69 | 93 |

The above data further illustrates the advantage of including both POET and $CaCl_2$ in the resin solution to improve the soiling and soil release properties of the treated fabrics.

## EXAMPLE IV

Samples of 100% polyester tricot were padded with aqueous solutions of various durable press resin compositions, dried for 10 minutes at 200° F., cured for 10 minutes at 350° F., scoured and redried, and finally evaluated for original whiteness and for soiling and soil re-

lease characteristics as described in Example I. The resin solutions evaluated were as follows:

Solution 1A:
10% dimethylol dihydroxy propylene urea 1.5% zinc nitrate catalyst
10% dimethylol dihydroxy propylene urea
1.5% zinc nitrate catalyst
Solution 1B:
Solution 1 plus 10% POET and 10% $CaCl_2$
Solution 2A:
10% dimethylol urea
1.5% zinc nitrate catalyst
Solution 2B:
Solution 3 plus 10% POET and 10% $CaCl_2$
Solution 3A:
10% cyclic urea-glyoxal type resin
Solution 3B:
Solution 5 plus 10% POET and 10% $CaCl_2$
Solution 4A:
10% dimethylol dihydroxy ethylene urea
1.5% zinc nitrate catalyst
Solution 4B:
Solution 7 plus 10% POET and 10% $CaCl_2$

The original fabric whiteness and the whiteness after soiling and after washing as measured on the D–40 instrument are given in the following table. An untreated fabric control is included for purposes of comparison.

TABLE IV

| Solution | Fabric whiteness | | |
|---|---|---|---|
| | Original | Soiled | Washed |
| 1A | 73 | 59 | 71 |
| 1B | 102 | 67 | 93 |
| 2A | 86 | 61 | 73 |
| 2B | 105 | 63 | 100 |
| 3A | 92 | 64 | 82 |
| 3B | 100 | 66 | 95 |
| 4A | 75 | 59 | 72 |
| 4B | 102 | 72 | 96 |
| Control | 100 | 64 | 85 |

The above data illustrates the excellent results obtained from incorporating POET and $CaCl_2$ in each of four different resin compositions. In each of the above four sets of data, the B sample which included the POET and $CaCl_2$ additives in the resin solution has better original, soiled, and washed whiteness than the A samples.

## EXAMPLE V

Samples of broadcloth, oxford cloth and poplin fabric composed of 65 percent polyester and 35 percent cotton were padded with various solutions of dimethylol dihydroxy ethylene urea resin. The treated fabrics were cured by pressing on a hot head press at 280° F. on a 5–10–5 cycle, followed by heating at 325° F. for 10 minutes. The compositions of the resin solutions evaluated were as follows.

Solution 1:
10% dimethylol dihydroxy ethylene urea
1.1% zinc nitrate catalyst
4.0% polyethylene base textile softener
Solution 2:
Solution 1 plus 20% POET
Solution 3:
Solution 1 plus 10% POET and 10% $CaCl_2$

The original fabric whiteness and whiteness after soiling and after washing were measured according to the procedure described in Example I. The results obtained are given in the following table. An untreated fabric control is included for purposes of comparison.



TABLE V

| Fabric | Solution | Fabric whiteness | | |
|---|---|---|---|---|
| | | Original | Soiled | Washed |
| Broadcloth | 1 | 74 | 63 | 73 |
| | 2 | 73 | 63 | 82 |
| | 3 | 91 | 66 | 86 |
| | Control | 102 | 64 | 75 |
| Oxford | 1 | 100 | 63 | 76 |
| | 2 | 87 | 64 | 85 |
| | 3 | 90 | 67 | 98 |
| | Control | 112 | 63 | 88 |
| Poplin | 1 | 86 | 63 | 73 |
| | 2 | 79 | 61 | 78 |
| | 3 | 80 | 60 | 85 |
| | Control | 90 | 65 | 83 |

The above data illustrate the improvement in final washed whiteness which is obtained by incorporating both the POET and $CaCl_2$ in the resin finish.

## EXAMPLE VI

In order to characterize the product of this invention, a sample of treated fabric was analyzed for calcium as finished and after scouring. The sample was an oxford cloth fabric composed of 65 percent polyester and 35 percent cotton which had been padded with an aqueous solution containing 10% dimethylol dihydroxy ethylene urea resin, 1.5% zinc nitrate catalyst, 10% POET, and 10% calcium chloride. A similar sample which had been treated with the resin only was included as a control. The following analytical results were obtained:

| Condition | Treatment | P.p.m., CaH |
|---|---|---|
| As finished | Resin only | 70 |
| | Resin plus salt | 13,700 |
| After scouring | Resin only | 79 |
| | Resin plus salt | 92 |

The above results show that the process of this invention provides a marked improvement in the soil and stain release and resist characteristics of the fabric treated by the process. In other words, fabrics so treated do not soil or stain as easily. Further, soil and stains are more easily removed from the fabric.

It is to be understood that the embodiments disclosed herein are merely illustrative of the invention and that other embodiment may be contempleted without departing from the spirit and scope of the invention.

What is claimed is:

**1.** The method of treating a fabric made of a material selected from the group consisting of polyester, cotton and rayon, or a blend thereof; comprising impregnating the fabric with an aqueous solution containing a resin selected from the group consisting of urea formaldehyde and melamine formaldehyde; said resin containing polyoxyethylene terephthalate and a salt selected from the group consisting of calcium chloride and potassium chloride; the amount of said resin being 5–20% by weight of the solution; the amount of polyoxyethylene terephthalate being 5–15% by weight of the solution; the amount of salt being 5–15% by weight of the solution; and heating the fabric for 1.5 to 15 minutes at a temperature in the range of 250 to 400° F. to cure the resin.

**2.** The method of claim **1** wherein the salt is calcium chloride.

**3.** The method of claim **1** wherein the salt is potassium chloride.

4. The method of claim 1 wherein the polyoxyethylene terephthalate is a reaction product of terephthalic acid with ethylene glycol and polyethylene glycol and the salt is calcium chloride.

5. The method of claim 1 wherein the polyethylene terephthalate is a reaction product of terephthalic acid with ethylene glycol and polyethylene glycol and the salt is potassium chloride.

6. A fabric treated as in claim 2.

7. A fabric treated as in claim 3.

8. A fabric treated as in claim 4.

9. A fabric treated as in claim 5.



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GEORGE F. LESMES, Primary Examiner

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8—115.6, 115.7, 116.3

# UNITED STATES PATENT OFFICE
# CERTIFICATE OF CORRECTION

Patent No. 3,539,286 Dated November 10, 1970

Inventor(s) Clarence A. Bowers and Graham Chantrey

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

Col. 7, delete lines 4 and 5.

Col. 7, line 58, "280° " should read ---380° ---.

SIGNED AND
SEALED
FEB 2 1971

(SEAL)
Attest:

Edward M. Fletcher, Jr.
Attesting Officer

WILLIAM E. SCHUYLER, JR.
Commissioner of Patents