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COMPOSITION FOR MAKING SOIL RELEASING, DURABLE PRESS FABRICS

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No Drawing. Application Nov. 15, 1967, Ser. No. 683,139, now Patent No. 3,521,993, dated July 28, 1970, which is a continuation-in-part of application Ser. No. 645,599, June 13, 1967. Divided and this application May 26, 1970, Ser. No. 40,717

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ABSTRACT OF THE DISCLOSURE

A composition for treatment of textiles to impart durable press and soil release properties. The composition comprises an aryl stearic acid, an aminoplast pre-condensate or other durable press reactant and a synthetic polymer which absorbs at least about five times its weight of water under alkaline conditions. Preferably the materials are dispersed in aqueous medium.

This is a division of prior U.S. application Ser. No. 683,139 filed Nov. 15, 1967, now U.S. Pat. No. 3,521,993 in turn is a continuation-in-part of U.S. application Ser. No. 645,599 filed June 13, 1967, now abandoned.

The present invention is concerned with the provision of resin- or reactant-treated textiles or the like which possess improved soil releasing properties.

The invention herein is particularly applicable to textiles of the durable-press types, whether "precured" or "postcured." As understood in the art, "precured" goods comprise fabrics which are cured before they are placed in final form. Such fabrics are usually processed by impregnating with a water-soluble aminoplast resin precondensate or like reactant, followed by drying and curing before cutting, sewing and/or otherwise putting the fabric into its final form. This technique may be used, for example, in the preparation of durable press sheets, shirting material or the like.

On the other hand, "postcured" textiles are those which are not cured until after they have been processed into final form, e.g. into a garment. In this case, the textile is impregnated with the resin precondensate or reactant and dried without curing, the goods being then put into the desired final form, e.g. by cutting and sewing into a garment and then pressing and curing to permanently fix the press. The latter approach, involving impregnation, drying without curing, garment formation, pressing and curing, is described in U.S. Pat. 2,974,432 and represents one way of preparing the durable press garments which have become popular in recent years. Durable press products may also be obtained by partially precuring the fabric during the drying operation and/or simultaneously pressing and curing. The invention herein may be used with any of these modifications, as well as with the more conventional procedures for preparing precured or postcured resin-treated fabrics.

One difficulty with conventional durable press fabrics, whether precured or postcured ,is their tendency to be stained by oil-borne and/or color bearing materials including, e.g. salad oils, motor oil, butter, gravy, lipstick,

hair oil and salad dressing, etc., as well as catsup, mustard, cranberry juice which might be considered waterborne and grass stains. This difficulty is at least partly due to the use of polyester fibers in durable press fabrics. Typically, polyester fibers are used together with cellulosic fibers such as cotton, because the cellulosic can be cross-linked with the resin to give the desired durable press characteristics but its tear strength is reduced by the process. Polyester fibers are rather strong and compensate for the loss of strength by the cellulosic. However, they are relatively sensitive to stains, particularly oil-borne stains, and when oily substances or color bearing materials are taken up by them, as well as by the resin and any other fibers, it is very difficult to remove them by laundering. The stains can be removed by dry cleaning but this is expensive and undesirable, particularly since durable press fabrics are otherwise well suited for home laundering and an important advantage thereof is that they retain their shape through laundering and need little, if any, ironing.

Numerous proposals have been made to improve the soil releasing properties of durable press fabrics, particularly with respect to stains of the type referred to above. Some of these proposals are undesirable because they require more than a single washing of the fabric to remove the stains. One process, which gives effective soil release in a single washing, is described in the copending application of Hinton et al., Ser. No. 604,649, filed Dec. 27, 1966, now abandoned, and a continuation-in-part thereof Ser. No. 681,092 filed Nov. 7, 1967, now abandoned, the subject matteer of which is incorporated herein by reference. According to said process, fabric which has previously been treated with textile resin or reactant is coated with a synthetic polymer, e.g. a copolymer of styrene and acrylic acid or ethyl acrylate and methacrylic acid of relatively high molecular weight which is water insoluble but absorbs at least about 550% by weight of water when immersed in an aqueous alkaline detergent solution having a pH of at least about 8 for 2 minutes at 140° F. This procedure gives good soil releasing properties with respect to a wide variety of stains but it is limited to use with precured fabrics, i.e. fabric which has already been impregnated with resin, dried and cured. A soil releasing effect can be obtained if treatment with the polymer precedes application of the resin precondensate or the reactant of the durable press type, but this undesirably affects the durable press properties. Application of the soil release copolymer of Ser. No. 604,649 to fabric containing the resin precondensate or reactant in the uncured state (i.e. postcured goods) is likewise undesirable. This is a serious limitation on the process of Ser. No. 604,649 since most durable press garments are presently postcured. Then, too, application of the process to precured goods has the disadvantage of requiring additional impregnating and drying steps which increase process costs.

The principal object of the present invention is to provide a process for improving the soil releasing properties of textiles, particularly durable press fabric, whereby prior difficulties are obviated. A more particular object of the invention is to provide a process of the type indicated which may be used with any type of durable press fabric, i.e., whether precured or postcured. Another specific object of the invention is to provide an improvement in the processing of durable press fabrics, as such,

or in garment form, whereby application of the resin precondensate or reactant used for durable press effects and the treatment for improving soil releasing properties are combined to minimize the number of steps involved. Other objects will also be apparent from the following detailed description of the invention.

The success of the invention is based on the finding that aryl stearic acids, preferably monocarbocyclic aryl stearic acids and especially phenyl stearic acid are unexpectedly effective in improving the soil release properties of textiles. In a particularly preferred embodiment of the invention it has been found that conventional aminoplast resin precondensates or other textile reactants and the water-absorbing or swellable polymers of Ser. No. 604,649 may be applied simultaneously to the fabric to obtain outstanding durable press effects and soil release properties provided the treating bath also includes phenyl stearic acid. The manner in which the acid functions to make this possible is not understood. It appears that the acid may react or otherwise cooperate with the aminoplast or textile reactant and the water-absorbing polymer to form a "matrix" polymer which gives the improved soil release properties. Whatever the explanation, however, the acid functions in such a way that the aminoplast precondensate or the like and water-absorbing polymer are compatible with each other and are able to exert their respective functions without interference to give the desired durable press effects and optimum soil release characteristics in the thus treated goods. The compatibility thus obtained means that the invention can be used whether the goods are to be precured or postcured with considerable simplification in processing techniques. For example, it is only necessary to impregnate the fabric with a single composition containing the precondensate, water-absorbing polymer and phenyl stearic acid, followed by drying, and precuring or postcuring as desired. Accordingly, the present process makes it possible to avoid the extra impregnating and drying steps of prior procedures and the accompanying expense. Furthermore, as opposed to presently known techniques, the invention gives not only a smooth drying fabric, but also provides the important function of soil release together with an improved degree of static control.

The stearic acid used herein may be represented by the following formula:

$$CH_3(CH_2)_8\underset{\displaystyle R}{\underset{|}{C}}H(CH_2)_7COOH$$

wherein R is an aromatic group. Preferably R is phenyl or naphthyl group, substituted optionally with alkyl, preferably lower alkyl groups, e.g. methyl. The aryl group also may be substituted with water solubilizing groups such as hydroxy, carboxy and sulfate. These may make it possible to omit an emulsifying agent from the treatment bath. It also is to be appreciated that isomers of the compound as shown, with the phenyl group disposed at any point from the second carbon in the stearic acid chain to the seventeenth, may be employed for present purposes. Phenyl stearic acid has been found particularly useful, but other aryl stearic acids, e.g. naphthyl stearic acid, xylyl stearic acid, and tolyl stearic acid, may also be used in lieu of, or in addition to the phenyl stearic acid. However, the latter is definitely preferred and gives the best results in terms of soil releasing properties. Phenyl stearic acid also provides the best or most pleasing "hand." Hydroxy substituted phenyl stearic acids are also useful, although they tend to cause yellowing of the goods.

The aryl stearic acid used herein may be prepared in conventional manner, e.g. by alkylation of benzene or the like with oleic acid in the presence of an acid activated clay or other acid catalyst.

As indicated, the water-absorbing polymer used herein may be any one or more of those described in Ser. No. 604,649 and the aforesaid continuation-in-part as soil release agents. These may be defined as polymers which absorb at least about five times the weight of water under alkaline conditions or, more specifically, at least about 50% by weight of water when immersed in an aqueous detergent solution for 2 minutes at 140° F. (pH about 8–12). Preferably, this polymer is an addition polymer of at least one ethylenically unsaturated monomer having one or more acid groups. Such monomers include, for example, acrylic acid, methacrylic acid, itaconic acid, maleic acid, fumaric acid and the like. Monomers which contain groups which readily hydrolyze in water to form acid groups also may be used, for example maleic anhydride. Preferably, the acid groups are all carboxylic acid groups. However, it is possible for a portion of them to be phosphoric acid (PO_4H_2) or sulfonic acid (SO_3H) groups as well, by using monomers such as styrene sulfonic acid and phosphoric acid ester of glycidyl allyl ether. The polymers may be manufactured by polymerizing the above monomers by any known means, for example with free radical or ionic catalysts. Alternatively, the polymers may be made by polymerizing an ethylenically unsaturated monomer such as acrylonitrile or an acrylic ester and then hydrolyzing or otherwise modifying the resulting polymer to convert the nitrile, ester or other groups to acid groups.

The polymers may contain relatively small amounts of monomers which do not include acid groups, for example olefins, e.g. ethylene and propylene, aromatic olefins such as styrene and various methyl styrenes, acrylic and methacrylic esters such as methyl methacrylate and ethyl acrylate, dienes such as butadiene and isoprene, vinyl halides, e.g. vinyl chloride and vinylidene chloride, other acrylic monomers such as acrylamide and acrylonitrile, and the like. In general, the proportion of such non-acid monomers should not exceed about 60 mole percent and preferably 40 mole percent. For maximum durability, it is preferred to use a system which leads to the crosslinking of the acrylic polymers, providing a matrix polymer with the other materials present.

Particularly useful materials for use herein as the water-absorbing or swellable component are: polyacrylic acid, acrylic acid or methacrylic acid copolymers for example, copolymers of styrene and acrylic acid, copolymers of itaconic acid and acrylic acid; and copolymers of ethyl acrylate and methacrylate acid; the copolymers of styrene, e.g. copolymers of styrene and maleic anhydride; and methacrylic acid and acrylic acid terpolymers such as terpolymers of methacrylic acid, butadiene and styrene; and terpolymers of monomethyl itaconate, acrylic acid and itaconic acid.

Preferably the water-absorbing polymer is one which absorbs at least about 1000% by weight of water. This is evidenced by very substantial swelling of the polymer although the polymer should not be soluble in alkaline solutions. The suitability of a polymer for use herein can be readily determined by measuring its water absorbing capacity or swellability. To do this, fabric treated with the polymer only is weighed, and the original weight of fabric is subtracted. The fabric is then immersed in detergent solution for two minutes at 140° F., blotted dry with paper towels and then weighed. A correction is made for the liquid absorption by the fabric itself by repeating the procedure with uncoated fabric. The swelling is equal to:

$$\frac{\text{weight gain in coating}}{\text{dry weight of coating}} \times 100$$

A typical detergent solution which may be used for this purpose is .15% Tide detergent in water. Tide comprises sodium lauryl sulfate 16%, alkyl alcohol sulfate 6%, sodium polyphosphate 30%, sodium pyrophosphate 17% and 31% sodium silicate and sodium sulfate combined.

The water absorbing (or swelling) characteristic of the soil release polymer is related to molecular weight. In general, low molecular weight polymers of the type described

above are water soluble whereas higher molecular weight polymers are insoluble in water but will swell. For the most part, suitable polymers will have a number average molecular weight of 1,000 to 3,000,000 although this is subject to some variation provided the desired degree of water absorption or swelling is realized without dissolution of the polymer.

Another factor affecting the water absorbing property or swellability of the polymer is the degree of crosslinking therein. The polymers used herein are essentially linear polymers. A certain degree of cross-linking may be introduced into the polymers during subsequent treatments. Although such cross-linking is not absolutely essential, it is desirable for durability. However, excessive crosslinking tends to connect the polymer molecules into a rigid three dimensional network which will not swell and this is not particularly useful for present purposes. Suitable crosslinking agents are formaldehyde, polyfunctional alcohols, formaldehyde amine precondensates, polyfunctional epoxides, etc. These may be included, as described, to improve durability.

Any of the aminoplast resin precondensates or other textile reactants conventionally employed for imparting durable press effects may be used herein. This includes formaldehyde and any of the water-soluble precondensates of formaldehyde with such amino compounds as urea, thiourea, cyclic ethylene ureas (e.g. dimethylol cyclic ethylene urea or dimethylol dihydroxy cyclic ethylene urea), melamine, ethyl carbamate, urons, triazones and triazines. Blocked isocyanates may also be effectively used.

Typically the invention is practiced by impregnating the fabric with an aqueous composition containing (1) the aminoplast resin precondensate or like reactants, (2) the water absorbing, swellable polymer as described above and (3) phenyl stearic acid. Wetting agents, plasticizers, softeners and the like may be employed as desired in conventional amounts. An appropriate catalyst for curing the aminoplast or like reactant should also be included and any of the standard catalysts may be used for this purpose, the ultimate selection depending on such factors as whether the fabric is to be precured or postcured. Typically suitable catalysts include magnesium chloride or nitrate or zinc chloride or nitrate; various amine hydrochlorides such as 2-amino-2-methyl-1-propanol hydrochloride, or triethanolamine hydrochloride; and ammonium salts such as ammonium chloride tartrate, citrate, formate, oxalate, nitrate or ammonium ethyl phosphate or ammonium dihydrogen phosphate or the like. These catalysts may be combined and/or catalysts modifiers may be added as necessary to achieve any desired effects, e.g. to increase or decrease catalyst activity.

Typically the composition used for impregnating the fabric (for example, by padding or spraying) will comprise, on a weight basis, from 5–20% aminoplast precondensate, usually 10–15%; 1–10% phenyl stearic acid, preferably 3–5%; 1–10% and preferably 2–5% soil releasing polymer; and 0.1–2.5% catalyst, balance water with the optional addition of wetting agents, plasticizers, softeners and the like as noted above. It will be recognized, however, that other proportions can be used, the optimum in any particular situation depending upon other operating factors, e.g. the nature of the fabric and its intended use. Advantageously, the components are simply added to water to make up the impregnating composition although it is preferred to emulsify the phenyl stearic acid in a mixture of water and xylene (or toluene) before adding same to the composition.

The amount of the composition applied to the fabric can be widely varied and is also dependent on such factors as the nature and construction of the fabric, its intended use, etc. Usually, however, wet pickup will fall in the range of 30–50% by weight of the fabric. This fabric is then dried, usually at 190–220° F. for 1–5 minutes, and cured at 300–350° F. for 1–15 minutes. Precured goods are usually cured for from 1 to 3 or 5 minutes and postcured goods are normally cured for somewhat longer times, generally from 10 to 15 minutes. The drying procedure provides a degree of partial cure, usually about 25%, and forms at least some crosslinking in the matrix polymer embodying the acid, the catalyst, the durable press resin, and the soil releasing polymer, because the essential components have been found resistant to wash off where a plural bath technique is employed.

In the case of postcured goods, the dried fabric may be cut into deired shape, sewn into a garment or other article, pressed on a hot head press or the equivalent and then cured. Whether precured or postcured, goods processed according to the invention demonstrate outstanding soil release and durable press properties as well as other essential characteristics such as hand and antistatic properties. On laundering, a single wash on a convention home washing machine using built detergents is sufficient to completely remove stains caused by substances such as salad or cooking oils, motor oil, butter, lipstick, hair oil, salad dressings, etc. Frequently the aqueous detergent or soap solutions have a pH of 7–12, although the invention may be useful with other soap or detergent solutions. The phenyl stearic acid somehow functions, presumably as a result of the formation of the matrix polymer, to permit the soil release polymer to absorb water and swell so as to facilitate removal of stains by the detergent while at the same time avoiding undesirable inter-reactions between the polymer, aminoplast and/or fabric which would prevent the aminoplast from providing permanent press effects. It is completely surprising that these two effects can be obtained together in the manner indicated.

The invention may be used to improve the soil release properties of any type of fabric, knitted, woven, or nonwoven, which is resin treated. It is of particular advantage in the case of durable press fabrics comprising blends of polyester and cotton fibers since these are especially susceptible to stains from oily substances. However, other types of fabrics made up entirely of natural or synthetic fibers, for example 100% polyester or 100% cotton, may also be effectively processed in the manner described herein using phenyl stearic acid to obtain improved soil release properties. Such other fibers include, in addition to polyester and cotton, glass, wool, rayon, cellulose acetates, polyamides, acrylics, polyolefins, separately or in admixture. Other nontextile substrates may also be usefully processed according to the invention where soil release is desired.

The invention is illustrated by the following examples wherein parts and percentages are by weight unless otherwise indicated.

EXAMPLE 1

A woven 65/35 polyester/cotton fabric was padded with the following formulation:

	Percent
Dihydroxy dimethylol ethylene urea	10
Phenyl stearic acid (water/xylene emulsion)	5
Copolymer of 2.7 moles methacrylic acid and 1 mole ethyl acrylate (molecular weight about 800,000 to 1,500,000)	5
Ammonium chloride	0.4 to 0.5
Water	Balance

Citric acid also may be used as a catalyst.

The wet pickup was about 40–45% on the dry weight of the fabric. The fabric was then dried at 200–220° F., for two minutes, cut, sewed into a garment leg; pressed on a hot head press at 300° F. (10 seconds) and postcured at 325° F. for 10 minutes.

The thus processed garment leg was then tested for soiling by spotting with various oily substances using the soil release test set forth below. The soil release was evaluated as Class 5, i.e. no visible oil stains remained, with only a single washing. The press was retained even after ten washings and other properties of the garment, e.g. softness and hand, were also outstanding.

The soil release test utilized herein was as follows:

The cured specimen, after conditioning for an hour, is soiled in different areas with corn oil, mayonaise, butter, lipstick, chocolate syrup, coffee and hair oil. The soiled sample is given a single washing in a home laundry agitator type washing machine using a low sudsing detergent (e.g. "Ad," pH about 10) in wash water at 140° F. After washing for 10 minutes, the sample is rinsed at 105° F., extracted and tumble dried at 150–170° F. After conditioning, the sample is placed on a black surface under a fluorescent light. The sample is visually rated under these conditions with numerical ratings as follows:

Class 5—No staining
Class 4—Slight, but not appreciable staining
Class 3—Noticeable staining
Class 2—Very notiecable staining
Class 1—Very extreme staining

EXAMPLE 2

Example 1 was repeated except that the fabric was precured by curing in the flat condition at 350° F. for 5 minutes. The resulting fabric remained wrinkle-free even after repeated washings and had a rating of 5 on the soil release test.

EXAMPLE 3

Example 1 was repeated except that a copolymer of styrene (2.5 parts) and acrylic acid (10 parts), molecular weight of about 5,000 was employed in lieu of the copolymer used in Example 1. Essentially similar soil release and durable press properties were obtained.

EXAMPLE 4

This example illustrates the application of the process to 100% polyester fabric:

The fabric was impregnated in an aqueous composition containing 5% phenyl stearic acid, 5% of the methacrylic acid/ethyl acrylate copolymer of Example 1 and 10% blocked isocyanate (i.e. Nopco D612, a polyisocyanate blocked with phenol), balance water. Wet pickup was about 35% on the dry weight of the fabric.

The fabric was then dried at 220° F., pressed and cured at 325° F., the isocyanate becoming unblocked during the cure to set the press. The press was retained after repeated launderings and the soil test rating was 5.

It is to be noted that other available blocked isocyanates (monomeric or polymeric) may be used in lieu of the Nopco 612 employed in the above formulation provided the unblocking thereof and reaction between the isocyanate and fabric readily occur at elevated temperatures which do detrimentally affect the fabric (e.g. 275–350° F.). Typically suitable for use herein are the aliphatic or aromatic polyisocyanates, e.g. toluene di- or tri-isocyanate, dimers or trimers thereof (such as shown in U.S. Pat. 2,801,244), hexamethylene diisocyanate or other alkylene polyisocyanates, blocked with phenol. The phenol-blocked polyisocyanates become unblocked at temperatures around 300° F., and at this temperature, the released isocyanate effectively crosslinks or otherwise reacts with the fabric to give the desired durable press effect.

While it is particularly advantageous, as exemplified above, to apply the phenyl stearic acid, aminoplast precondensate or other reactant and soil release polymer from a single bath, the invention also contemplates the possibility of using a two bath technique wherein the phenyl stearic acid is first applied to the fabric, followed by drying and subsequent application of the aminoplast and soil release polymer. The two bath embodiment is illustrated by the following example:

EXAMPLE 5

A woven garment fabric made from yarn comprising 65% polyethylene terephthalate fibers and 35% cotton fibers (by weight) as in Example 1 was padded with an aqueous emulsion of 3% phenyl stearic acid. The fabric was heated at 325° F. for about one minute whereby the fabric was dried with apparent insolubilization of the phenyl stearic acid and the formation of a film thereof on the fabric.

The thus treated fabric was then impregnated with an aqueous solution of the following composition:

	Percent
Copolymer of about 2.7 moles of methacrylic acid and about 1 mole ethyl acrylate (molecular weight about 300,000)	8
Dihydroxydimethylol ethylene urea	10
Ammonium chloride catalyst	.25
Polyvinyl methyl ether	1
Water	Balance

Wet pickup amounted to about 45% on the weight of the fabric.

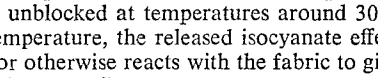
The fabric was thereafter dried by heating at about 275° F. for 4–5 minutes. This resulted in a partial precure (equivalent to about 30–40% fixed solids). The fabric was then cut and sewed into garments, pressed and then postcured in an oven at about 325° F. for about fifteen minutes.

The resulting garment was soft and otherwise demonstrated an excellent hand and possessed outstanding press retention even after repeated washings. When subjected to the soil release test referred to above, the oil stains were immediately and completely removed to give a rating of 5.

It will be recognized that various modifications may be made in the invention described herein. In its broadest aspects, the invention contemplates the treatment of textile materials with an aryl stearic acid, particularly phenyl stearic acid, as such or in combination with a water-absorbing swellable polymer as described to improve soil release properties whether or not durable press properties are involved. It is also contemplated that the treatment with phenyl stearic acid according to the invention may be used to improve the soil release properties of garments or fabrics which have previously been given durable press treatments. Other modifications will also be apparent. For example, the treatment according to this invention can also be applied with useful effect to textile substances generally, not requiring the presence of cellulose, and to nontextile substrates, such as wood and concrete, to render them soil releasing. Hence, the scope of the invention is defined in the following claims wherein—

What we claim is new:

1. A composition for rendering textile materials soil releasing which comprises an aryl stearic acid selected from the group consisting of aryl stearic acids having the formula



where R is aryl and isomers thereof wherein the aryl group is disposed at any point from the second to the seventeenth carbon atom, a durable press textile reactant selected from the group consisting of formaldehyde, water-soluble precondensates of formaldehyde with amino compounds and blocked isocyanates, and a synthetic polymer which absorbs at least about five times its weight of water under alkaline conditions, said polymer being an addition polymer of at least one ethylenically unsaturated monmoer having one or more acid groups.

2. A composition according to claim **1** wherein said polymer is a methacrylic acid/ethyl acrylate copolymer.

3. A composition according to claim 1 wherein the textile reactant is a blocked isocyanate.

4. A composition according to claim 1 in the form of a cross-linked matrix polymer.

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