

[54] MULTI-STEP IN SITU POLYMER FORMATION TO PRODUCE FABRIC HAVING IMPROVED SOILING CHARACTERISTICS

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[*] Notice: The portion of the term of this patent subsequent to Apr. 9, 1985, has been disclaimed.

[22] Filed: Apr. 17, 1967

[21] Appl. No.: 631,165

[52] U.S. Cl.8/115.5, 8/115.6, 8/115.7, 8/116, 8/116.3, 8/116.2, 8/127.6, 8/128, 8/DIG. 4, 8/DIG. 21, 38/144, 2/243, 117/93.31, 117/138.8 A, 117/138.8 F, 117/138.8 N, 117/139.5 A

[51] Int. Cl.D06m 15/36, D06m 15/58, D06m 15/72

[58] Field of Search8/DIG. 18, 116, 116.3, 115.7, 8/116, 115.5, 115.6; 117/93.31, 161, 139.4, 143, 138.8

[56]

References Cited

UNITED STATES PATENTS

Table with 3 columns: Patent Number, Date, and Inventor/Reference. Includes entries like 3,097,960 7/1963 Lawton et al. 8/DIG. 18, 3,101,276 8/1963 Hendricks 8/DIG. 18, etc.

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[57]

ABSTRACT

A process for improving soil release characteristics of textile material which comprises applying thereto a polymerizable compound and forming in situ a synthetic acid polymer comprising at least 10 weight percent acid calculated as acrylic acid, and the product produced by this process. Preferably, a textile resin and a textile resin catalyst also are applied to the textile material and the treated textile material is subjected to resin curing conditions.

17 Claims, No Drawings

MULTI-STEP IN SITU POLYMER FORMATION TO PRODUCE FABRIC HAVING IMPROVED SOILING CHARACTERISTICS

BACKGROUND OF THE INVENTION

The textile industry during the past decade has, as a whole, made important technological advances in the chemical finishing of textile fabrics. Numerous processes have been developed for imparting minimum care characteristics to garments and articles prepared from specially treated textile fabrics. Exemplary of such advances are the wash and wear fabrics, hereinafter referred to as precured fabrics, and the durable press fabrics, hereinafter referred to as post cured fabrics. These characteristics generally have been imparted to textile fabrics by the application of resinuous materials. The resinuous materials are applied to the fabric and are later crosslinked to the fabric by the action of a suitable catalyst. Depending upon the time at which the crosslinking reaction occurs, either a wash and wear fabric or a durable press fabric is produced. The precured fabrics are those for which the crosslinking reaction has occurred prior to transformation of the fabric into a garment or other article of commerce. Post cured fabrics are those fabrics which are subjected to the crosslinking reaction subsequent to the transformation of the fabric into a garment or other article of commerce.

Tremendous effort has ensued towards achievement of a garment containing synthetic and naturally occurring fibers such that creases in the garment are very durable and are not appreciably affected by wear or cleaning processes. In other words, after repeated washings and/or dry cleaning, the creases remain in the garment in a substantially unaltered condition and further treatment of the garment, i.e., pressing, is not required for maintenance of the crease. Likewise, much effort has been expended towards the attainment of good wash-and-wear fabric.

Additionally, further research has been directed to the attainment of a garment having improved soil release properties. Numerous of the synthetically produced fibers that are presently being incorporated in blends with naturally occurring fibers have a propensity to accept and retain oily grime and dirt. Accordingly, when the garment is being worn the soil and/or oily materials accumulate on the garment and settle in the fabric. Once the garment becomes soiled, it is then subjected to a cleaning process for removal of the dirt and/or oily deposits, and only a dry cleaning process will successfully clean the garment.

The cleaning process normally employed, however, is washing in a conventional home washing machine by the housewife. During a wash cycle, it is virtually impossible to remove the soil and/or oily stains from the garment and, secondly, assuming that the undesirable materials are removed from the garment or a fairly clean garment is being washed, soil remaining in the wash water is redeposited onto the garment prior to the end of the wash cycle. Hence, when the garment is removed from the washing machine and subsequently dried, it has not been properly cleaned. Such a condition, heretofore unavoidable, is quite disadvantageous in that the garment after being worn never again assumes a truly clean appearance, but instead tends to gray and/or yellow due to the soil and/or oily materials deposited and remaining thereon. Further use and washing of the garment increases the intensity of the graying to the point that ultimately the garment is unacceptable for further wear due to its discoloration. The process of the present invention solves the soiling problem as hereinafter described.

In attempting to solve the problem of soiling in the synthetic fabrics and blends containing synthetic fibers, a substantial amount of research has been conducted and numerous patents have issued as a result thereof. None of these patents, however, disclose subject matter as relevant to the problem as is instantly set forth herein. Strong basis for this fact is evidenced by the publicity being given to products exhibiting even slight improvement in soil release and/or soil redeposition. Antisoil-

ing research has been directed along two general avenues, one of which utilizes inorganic materials and the second employing organic materials. Set forth below is a brief summary of prior effects.

U. S. Pat. No. 2,999,774 to Schappel features the utilization of silica particles and a salt of a multivalent metal for the purpose of rendering a fabric soil resistant. U. S. Pat. No. 2,734,835 to Florio et al. employes at least two hydrous stable metal oxides selected from aluminum, silica, titanium, beryllium, cerium, cobalt, germanium, manganese, tin, zinc and zirconium. U. S. Pat. No. 3,089,778 to Pierce et al. teaches the utilization of a water insoluble basic aluminum salt having an ultimate particle size of less than 0.5 microns. U. S. Pat. No. 2,992,943 to Coover et al., while not purely related to inorganic materials is directed to prevention of dry soiling only. In other words, the Coover et al. treatment dictates the use of a water-soluble compound (an alkyl titanate and an organic solvent) and therefore to obtain the desired soil resistant properties only a dry cleaning process may be employed.

The organic approach to the soiling problem of synthetic fiber containing fabrics includes the following patents and their teachings. It should be noted, however, that some of the patents incorporated in the following group are not per se directed to reducing the soil propensity of the synthetic fiber containing fabric.

U. S. Pat. No. 3,236,685 to Caldwell et al. renders a fabric antistatic and soil-resistant by coating a fabric with a solution or solutions containing a polymeric acid defined as containing —COOH, —SO₃H and/or —PO₄H₂ groups. Additionally, a compound containing a polyol or a compound having incorporated therein epoxide groups is included which under proper conditions reacts with the acid to form an ester. U. S. Pat. No. 3,152,920 also to Caldwell et al. is a complement of the above patent wherein, instead of reacting the polymeric acid with a polyol or an epoxide, the polymeric acid is reacted with the reaction product of a polyol and a polyisocyanate. U. S. Pat. No. 3,125,405 to Gardon is directed to the manufacture of a permanent press garment. N-methylol acrylamide is applied to the fabric with a free radical acid catalyst and the N-methylol acrylamide is crosslinked with the cellulose molecule. Additionally, extra monomers and polymers as set forth in the patent may be incorporated in the treating solution. U. S. Pat. No. 3,246,946 to Gardon likewise is directed to the production of durable press garments. N-methylol acrylamide is employed in conjunction with one or more condensates of an aldehyde and a free radical acid catalyst whereby the reactants are crosslinked with the cellulose molecule. Extra monomers and polymers may be added to the treating solution. U. S. Pat. No. 3,090,704 to Collins et al. is directed to a terpolymer for rendering the fabric soil resistant. The terpolymer consists of (1) a compound having incorporated therein a crosslinking component, (2) a compound having incorporated therein an anionic component, e.g., an alkali metal salt of an aromatic sulfonic acid, and (3) a compound having a component therein that contains a strong nonionizable, non-hydratable permanent or induced dipole. U. S. Pat. No. 2,876,141 to Matthews employs a solution containing (1) mineral oil, (2) base cordage oil, (3) oleic acid, and (4) a cationic wetting agent, e.g., trimethyl-β-oleamidoethyl ammonium sulfate in an effort to improve the soil resistance of the fabric treated.

The above brief abstracts are set forth to provide an indication of prior research effort directed to attaining a soil resistant fabric or a fabric having soil release properties. The problem heretofore confronted with fabrics including synthetic fibers has been that the synthetic fibers while hydrophobic are oleophilic and whereas oil and grime may become embedded in the fiber, its hydrophobic properties prevent water from entering the fiber to remove the contaminants therefrom. The efforts of this invention have been directed to the modification of the properties of synthetic and/or natural fibers in such a manner that the soil and oily contaminants may be easily removed.

Additionally, by incorporating the process of the present invention with that of a process to render a garment resistant to creasing, a garment is produced that has both durable press and soil release properties. In other words, the ultimate garment is superior both for the consumer and for the housewife who is confronted with the problem of rendering the garment clean for further wearing.

In view of the above comments, it should be evident to one skilled in the art that the problem confronted has been that of rendering a garment clean if the garment contains synthetic and/or natural fibers as described herein. Accordingly, by virtue of the teachings of the present invention, the problems historically present with the use of garments having incorporated therein both cellulosic fibers and synthetically produced fibers are alleviated.

It is therefore an object of the present invention to provide a substrate having improved soil release properties.

Still another object of the present invention is to provide a process for treating textile material whereby said material easily releases soil when contacted with a detergent solution.

Still further another object of the present invention is to treat textile material in such a manner that after said material is soiled and subjected to washing, less soil and grime from the wash water will be redeposited thereon.

A further object of the present invention is to provide a durable press fabric having improved soil-release properties.

Another object of the present invention is to provide a process for treating a fabric in such a manner that it has both durable press and soil release properties.

Still another object of the present invention is to treat fabric in such a manner that after a garment produced therefrom is soiled and subjected to washing, soil and grime from the wash water will not be redeposited onto the garment.

Still further another object of the present invention is to provide a treatment for fabric such that garments produced therefrom will not become discolored due to repeated wearing and washing.

Another object of the present invention is to treat fabric in such a manner that a garment produced therefrom has excellent wash-and-wear and soil release properties.

These and other objects may be readily seen from the following detailed description of the present invention.

Generally speaking, the present invention is directed to a process for treating textile material comprising applying thereto a polymerizable compound and forming a synthetic acid polymer, said polymer containing at least 10 weight percent acid calculated as acrylic acid.

Soil removal ability is improved on any textile material when the acid polymer is formed by polymerization in situ. Suitable textile materials include synthetic polymers, cotton, wool, mixtures of the above, etc. Products made from these materials include without limitation, textile fabric wall coverings; lamp shades, automobile seat covers; automobile upholstery, e.g., door panels, overhead liners, etc.; upholstery for furniture; clothing; apparel accessories, e.g., ties, fabric belts, scarves, hats, etc.; canvas products, e.g., tents, folding cots, etc.; draperies; throw pillows; hassocks; sporting goods; fabric garment bags and luggage; fabric handbags; fabric shoes or shoes made from synthetic materials; linens; book covers; mattress covers; stuffed toys; hammocks; deck chairs, etc.

Textile materials which may be treated according to the process of the invention include those in which the anhydroglucose molecules are chemically substantially unmodified, e.g., cotton, paper, linen, jute, flax, regenerated cellulose fibers, including viscose rayon, in the form of staple, yarn and fabrics. This invention is directed primarily and preferably to textile fabrics either knitted, woven, or non-woven, preferably woven. However, the advantages of this invention can be achieved by treating the fibers, yarns, or threads employed to produce these fabrics.

Moreover, and more specifically, the process of the present invention is preferably used for treating textile materials containing both cellulosic and non-cellulosic fibers, especially if

the non-cellulosic fibers have minimum care characteristics of their own. For example, the fabrics treated may be formed from a mixture of polyester, such as poly(ethylene terephthalate), polyamide such as poly(hexamethylene adipamide) or acrylic fibers, such as polyacrylonitrile, and copolymers containing at least about 85 percent combined acrylonitrile filaments or fibers, with cotton or rayon. It should be pointed out, however, that textile material containing only non-cellulosic fibers such as those listed above is also within the scope of the present invention.

The soil release properties of pure cellulosic fiber fabrics are much better than those of synthetic fiber containing fabrics, e.g., polyester fibers, in that the synthetic polyester fibers are hydrophobic and thus prevent the ingress of water that is necessary for cleaning the fabric and also possess an electrical charge that attracts soil particles. The present invention is therefore most primarily directed to fabrics containing a substantial portion of synthetic fibers, but is not limited thereto. Instead, it has been determined that the present process can be very successfully employed with cellulosic fabrics, fabrics containing synthetic fibers and cellulosic fibers, and fabrics containing only synthetic fibers.

In many instances, when the substrate is a textile material, an amino-plast textile resin or a vinyl monomer with dual functionality will also be applied with the polymerizable compound. Very unexpectedly, it has been observed that when this combination is applied to the textile material followed by subjecting the material to textile resin curing conditions, improved soil release is realized.

Hence, the present invention is also directed to a process for treating a textile material by applying thereto an aminoplast textile resin, a textile resin catalyst and a polymerizable compound and forming a synthetic acid polymer from said compound, said polymer containing at least 10 weight percent acid calculated as acrylic acid and curing of the textile resin.

The term "textile resin" according to the present invention includes both monomers and polymers which when applied to a textile material and reacted under proper conditions undergo polymerization and/or condensation and are transformed to the thermoset state. Textile resins that may be employed when practicing the present invention are the aminoplast resins. These nitrogen containing resins when applied to a textile material in the presence of a catalyst at temperatures of from about 100° C. to about 300° C. are transformed into the thermoset state. The cured textile resin on the textile material affords the textile material a durable press and/or wrinkle resistant characteristic.

Exemplary of the aminoplast textile resins that may be employed according to the present invention are the urea formaldehydes, e.g., propylene urea formaldehyde, dimethylol urea formaldehyde, etc.; melamine formaldehydes, e.g., tetramethylol melamines, pentamethylol melamines, etc.; ethylene ureas, e.g., dimethylol ethylene urea, dihydroxy dimethylol ethylene urea, ethylene urea formaldehyde, hydroxy ethylene urea formaldehyde, etc.; carbamates, e.g., alkyl carbamate formaldehydes, etc.; formaldehyde-acrolein condensation products; formaldehyde-acetone condensation products; diureas, e.g., trimethylol acetylene diurea, tetramethylol-acetylene diurea, etc.; triazines, e.g., dimethylol-N-ethyl triazone, N-N'ethylene-bis dimethylol triazone, halotriazines, etc.; halo-acetamides, e.g., N-methylol-N-methylchloroacetamide, etc.; urons, e.g., dimethylol uron, dihydroxy dimethylol uron, etc., and the like. Mixtures of aminoplast textile resins are also within the scope of the present invention.

Further exemplarity of the vinyl monomers having dual functionality within the scope of the present invention are acrylamides, e.g., N-methylol acrylamide, N-methylol methacrylamide, N-methylol-N-methacrylamide, N-methylolacrylamide, N-methyl methylene-bis-(acrylamide), methylene-bis-(N-methylol acrylamide), etc.; haloethylene acrylamide; and compounds which conform to the following structural formulae. In each of the following formulae the variables may be selected as follows:

R¹: hydrogen, lower alkyl or residue of saturated or unsaturated aldehyde

R²: hydrogen, lower alkyl or -CX-CR³=CHR⁴

R³: hydrogen or methyl

R⁴: hydrogen or lower alkyl

R⁵: hydrogen, lower alkyl, or CHR¹OR⁴, at least one R⁵ being CHR¹OR⁴

R⁶: lower alkyl or hydroxy alkyl

R⁷: hydrogen, hydroxyl or lower alkyl

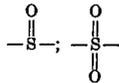
R⁸: hydrogen, lower alkyl, alkylol or alkenol

X: sulfur or oxygen

and where

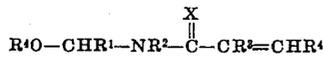


may have substituted therefor

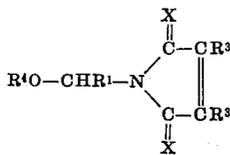


or sulfonium if desired.

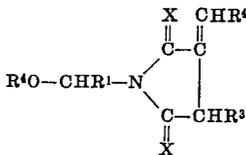
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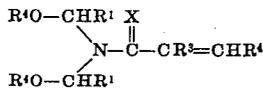
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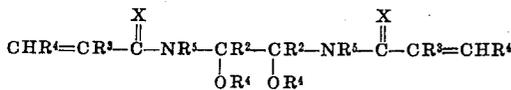
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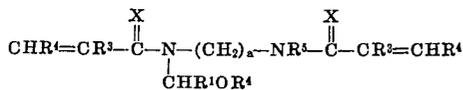
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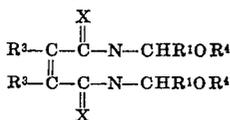


VI.

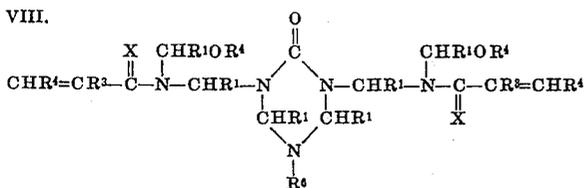


where a is a whole integer from 1 to 6

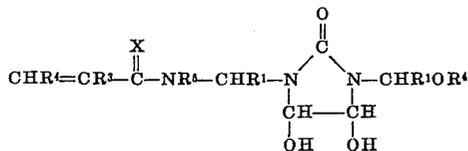
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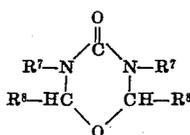
VIII.



IX.



X.



The amount of textile resin or vinyl monomer with dual functionality applied to the fabric is primarily determined by the ultimate use of garments or articles prepared from the fabric. Very small amounts will afford some improvement and

5 large amounts even greater improvements, but the larger amounts generally adversely affect the hand of the fabric. Hence, the amount employed is preferably that which will afford good crease retention and flat dry properties while not adversely affecting the hand. For the purposes of the present invention, the amount of textile resin or vinyl monomer in the pad bath may vary between about 2 and 30 percent. The proportion present on the fabric should be in the range of about 2 to 20 percent based on the dry weight of the fabric and preferably in the range of about 4 to 9 percent.

15 Catalysts employed within the scope of the present invention depend upon the specific textile resin or vinyl monomer that is applied to the textile material. For instance, if the textile resin has a functional group that is reactive under acidic conditions, then an acid catalyst is used. Likewise, when a functional group is present that is reactive under alkaline conditions, then a base catalyst is used. Furthermore, both acid and base catalysts may be used when both type functional groups are present in the textile resin. In this instance, the catalyst may be added separately or together. When they are added together, one must be a latent catalyst, i.e., one that will not initiate its reaction during the opposite type reaction, but may be activated subsequently under proper catalytic conditions.

30 The catalysts useful in activating the acid or base reactive groups are those conventionally used to activate the reaction of textile resins containing the same group for reaction with hydroxy groups of cellulose. Preferably, latent acid or base acting catalysts are utilized, that is, compounds which are acidic or basic in character under the curing conditions. The most common acid acting catalysts are the metal salts, for example, magnesium chloride, zinc nitrate and zinc fluoroborate and the amino salts, for example, monoethanolamine hydrochloride and 2-amino-2-methyl-propanol nitrate.

40 The base acting catalyst preferably is a compound which does not initiate substantial reaction between the base reactive group and hydroxy groups of cellulose under normal acid conditions, but does initiate substantial reaction under prescribed conditions, such as elevated temperature or some other activating means, as through use of another chemical compound. For example, an alkali metal sulfite can be padded onto the fabric and be decomposed into strongly basic alkali metal hydroxide by including small amounts of formaldehyde in the steam used for curing.

50 The latent base acting catalyst utilized herein preferably comprises alkali-metal salts, such as alkali-metal carbonates like sodium carbonate, which is neutral to mildly alkaline, for example, pH of about 8.5 on the fabric but decomposes at temperatures in excess of about 80° C. to form the stronger base sodium oxide which will initiate substantial reaction at the elevated temperatures utilized during curing. Sodium carbonate may be utilized if desired since the pH in the fabric produced by this compound in normal conditions is generally insufficient to initiate the desired degree of reaction under normal temperature conditions.

If fabrics containing a base reactive group are maintained at pH levels above about 10, however, degradation occurs, so that essentially neutral or mildly alkaline catalysts are preferred when base reactive compounds are utilized.

65 Additional base acting catalysts include potassium bicarbonate, potassium carbonate, sodium silicate, alkali metal phosphates, such as sodium or potassium phosphates, barium carbonate, quaternary ammonium hydroxides and carbonates, for example, lauryl trimethyl ammonium hydroxides and carbonates and the like.

70 The amount of catalyst to be utilized is that conventionally used in activating the reaction, for example, up to about 15 percent by weight of an acid acting catalyst in the application bath with the preferred range being from about 1 percent to

about 7 percent. A preferred range for the base acting catalyst is again the conventional amount and is generally between about 0.2 percent to about 16 percent, preferably about 2 to 16 percent. The amount of catalyst to be utilized will further depend in part on the temperature at which the reaction is conducted and the amount of catalyst consumed in the reaction. For example, when base catalysts are utilized and if a highly acidic group is released during the reaction, the amount of base applied to the textile material should be at least sufficient to provide an excess of base in addition to that which is consumed by the highly acidic group.

The term "soil release" in accordance with the present invention refers to the ability of the fabric to be washed or otherwise treated to remove soil and/or oily materials that have come into contact with said material. The present invention does not per se prevent the attachment of soil or oily materials to the fabric, but hinders such attachment and renders the heretofore uncleanable fabric now susceptible to a successful cleaning operation. While the theory is still somewhat of a mystery, soiled, treated fabric when immersed in the detergent containing wash water experiences an agglomeration of the oil at the fabric surface. This water is basic in nature and it has been determined that soil release is best realized in wash water that is basic in nature. These globules of oil are then removed from the fabric and rise to the surface of the wash water. This phenomenon takes place in the home washer during continued agitation, but the same effect has been observed even under static conditions. In other words, a strip of polyester/cotton fabric treated according to the process of the present invention and soiled with crude oil, when simply immersed in a detergent solution will lose the oil without agitation. The oil just balls up on the fabric, dislodges therefrom, and rises to the surface of the solution.

An added feature of the present invention is the prevention of soil redeposition from the wash water. One of the greatest disadvantages of the synthetic polymers is the feature that even after removing the soil by washing, there is the continued danger that the soil will be redeposited onto the fibers from the wash water before the garment is removed therefrom. It has been observed that the soil release ability of the presently treated fabric diminishes after repeated washings. Even after the ability to remove soil from the fabric has diminished, however, the observation has been made that the prevention of redeposition of soil from wash water remains potent. This phenomenon likewise is unexplainable, but it has been established that the troublesome soil is negatively charged and presumably there remains enough acid on the fabric to repel the negatively charged soil.

Numerous of the textile materials that may be treated according to the process of the present invention may not be feasibly removed from their environment and washed in a washing machine. Further, there are also textile materials that may be treated which when subjected to the action of a washing machine are adversely affected either in structure or in looks. Articles within these classes may still be easily cleaned in place or otherwise by scrubbing the soiled area lightly with a solution of a commercial detergent and water.

The soil release polymer of the present invention which will also be hereinafter referred to as "acid polymer," is formed in situ on the textile material from the polymerizable compound applied thereto. This acid polymer may be formed from a large number of compounds. The acid polymer formed advantageously is capable of forming a film around the fibers that constitute the textile material. Further, the film may have hydrophilic properties and be at least partially insoluble in water. The film, if water soluble, would, of course, be easily washed from the fabric. The polymer likewise may be water soluble if the textile material is such that the soil removal is only required once. Acid content of the polymer is likewise important and at least 10 weight percent of the acid polymer should be acid calculated as acrylic acid and preferably at least 20 weight percent. It has further been observed that all of the acid polymers formed in situ that afford soil release have a

carbon atom to acid group ratio in the repeat group in the range of 2:1 to 30:1, and that an air dried film cast therefrom has a water of imbibition of at least 89 percent.

Synthetically produced acid polymers within the scope of the present invention may be formed in situ on the textile material from any of the polymerizable organic acids, i.e., those having reactive points of unsaturation. These polymerizable compounds may be acids, or mixtures of an acid and other monomers copolymerizable therewith so long as at least 10 weight percent acid monomer is present in the polymer formed. Exemplary of polymerizable acids that may be used, are 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 capable of copolymerizing with the acids and which will not detrimentally affect the soil release properties of the polymer. Suitable monomers include, esters of the above acids prepared by reacting the particular acid with an alkyl alcohol, e.g., ethyl acrylate, methyl acrylate, propyl acrylate, isopropyl acrylate, butyl acrylate, methyl methacrylate, ethyl methacrylate, 2-ethyl hexyl 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. In all of the polymers formed from the above listed monomers, there should be at least 10 weight percent acid calculated as acrylic acid. Furthermore, salts of the acid polymers, e.g., sodium, potassium, lithium, ammonium, etc., will afford the desired soil release characteristics.

Examples of some of the synthetic acid polymers that may be formed in situ from ethylenically unsaturated monomers 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-ethyl hexyl acrylate:acrylic acid
 acrylamide:acrylic acid
 butyl acrylate:acrylic acid:acrylamide
 ethyl acrylate:acrylic acid:N-methylol acrylamide
 ethyl acrylate:acrylic acid:styrene
 ethyl acrylate:acrylic acid:hydroxy propyl 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:hydroxy ethyl methacrylate
 hydroxy ethyl methacrylate:acrylic acid:acrylamide
 butyl acrylate:ethyl acrylate:acrylic acid and the like.

Some acid polymer systems work better than others, however, and these are preferred. Examples of the preferred acid polymers include (1) copolymers of an acrylic ester and an acrylic acid that are prepared by polymerizing on the textile material a co-monomer mixture of from about 20 to 80 parts of the acrylic ester and about 20 to 80 parts of an acrylic acid; (2) copolymers of ethyl propyl or isopropyl acrylate and acrylic acid wherein the copolymers are prepared by polymerizing a monomer mixture of from about 40 to 80 parts of the acrylate and about 20 to 60 parts of acrylic acid; (3) copolymers of butyl acrylate and acrylic acid prepared by polymerizing a co-monomer mixture of from about 30 to 70 parts of butyl acrylate and about 30 to 70 parts of acrylic acid; (4) copolymers of 2-ethyl-hexyl acrylate and acrylic acid prepared by polymerizing a co-monomer mixture of from about 10 to 40 parts of 2-ethyl hexyl acrylate and about 60 to 90 parts of acrylic acid; (5) copolymers substantially identical to the ones listed above

with the exception that methacrylic acid is substituted for acrylic acid and the esters are methacrylates instead of acrylates; (6) a copolymer of ethyl acrylate and itaconic acid prepared by polymerizing a monomer mixture comprising about 70 parts ethyl acrylate and about 30 parts itaconic acid; (7) copolymers of the acrylic acid set forth above wherein the acrylates are substituted by methacrylates; (8) a copolymer of acrylamide and acrylic acid prepared by polymerizing a monomer mixture comprising about 10 parts acrylamide and about 90 parts acrylic acid; and (9) terpolymers comprising ethyl acrylate, acrylic acid and acrylamide prepared from monomer mixtures of ethyl acrylate, at least 10 parts acrylic acid and up to 20 parts acrylamide.

As the acid polymer is formed in situ on the textile material, soil release ability is created. For unknown reasons, further treatments and/or ingredients will enhance the soil release ability of the textile material. If the textile material having the acid polymer thereon is subjected to textile resin curing conditions, the durability of the soil release ability is enhanced. Likewise, the presence of a textile resin catalyst during the textile resin curing conditions further improves soil release durability. Furthermore, there may be some crosslinking between the cellulose molecules and the acid polymer or there may be just an enhanced physical bond between the textile resin and the acid polymer above and beyond their reactivity.

Soil release polymers, like the textile resins, give some improvement at very low levels on the fabric. Accordingly, as the amount of soil release polymer is increased, the ability of the fabric to release soil increases. Thus, the upper limit on the amount of soil release polymer is determined by economics and resulting adverse effects on the fabric, e.g., the hand of the fabric. Furthermore, practically speaking there is a set range of soil release polymer dictated by commercial success. Advantageously, there should be from about 1 to 20 weight percent of acid polymer on the substrate, based on dry weight, and preferably about 2.5 to 10 weight percent.

The materials used to impregnate the textile material according to the present invention are not limited to including only the materials heretofore mentioned, e.g., textile resin, vinyl monomers with dual functionality, catalyst and polymerizable compound to form the acid polymer. In addition, other ingredients may be employed such as, for example, emulsifying agents, wetting agents, softeners, etc., and numerous other compounds that enhance the physical characteristics of the fabric. The materials may be applied to the textile material in any suitable manner. For instance, application of the polymerizable compound to the fabric with a roller is preferred because of the soil release characteristics which can be achieved without curing when concentrated monomer is applied. The ingredients may also be sprayed on as liquids; the textile material may be treated with vapors of the compounds if convenient or the textile material may be dipped, etc.

In general, the applicator systems provide from 5 to 100 weight percent wet pickup by the fabric. When the mixture is padded onto the fabric, good results are obtained by providing a wet pickup of from 40 to 60 weight percent. With a roller, the wet pickup is usually between 5 and 50 weight percent and preferably 10 to 20 weight percent.

When an aminoplast textile resin or vinyl monomer with dual functionality is applied to the textile material along with the polymerizable compound, advantageously they are applied sequentially. Insofar as separate application is concerned, where the textile resin or vinyl monomer is applied first and the polymer formed or cured and the polymerizable compound is added separately thereafter, soil release ability of a higher level is achieved as compared with simultaneous application or the separate addition where both are present during curing.

According to the desires of the individual, and the dictates of the ultimate product, separate or simultaneous application of the textile resin and the polymerizable compound may be employed. For instance, when treating a textile fabric which is

to be converted into work clothes, it would be desirable to have as durable a finish as possible so that the soil release properties will be as long lasting as possible. For these items it is preferred to first apply the textile resin and separately after curing of the textile resin apply the polymerizable compound and subsequently form the soil release polymer. On the other hand, where the ultimate article of manufacture is not one that will be washed or cleaned on a weekly basis, for example, upholstery for automobiles, seat covers, wall coverings, etc., either a simultaneous addition or a separate addition where the polymerizable compound is added and the polymer formed first may be used. It must be emphasized, however, that under the latter conditions the soil release properties are less desirable than those attained by the aforesaid sequential application with the textile resin being cured first.

Advantages afforded by the process of the present invention are available for textile materials treated in almost any form, e.g., fibers, yarns, threads, fabrics or the ultimate product, e.g., a garment, etc. The presensitizing embodiment, i.e., the textile resin treatment, when employed, is most advantageously conducted on textile material in the fabric, etc., form.

Garments made from the fabrics treated according to the process of the present invention require no steps other than normal for the preparation of the conventional durable press garments. In other words, the garment may be folded and pressed on conventional equipment, for example, a Hoffman press. The pressing cycle utilized is standard in the industry and generally involves pressing of the garment for a short period of time, followed by a curing operation in an oven. Alternatively, the garment may be set in a desired configuration under hot, dry conditions, such as by hot pressing without steaming, for example at temperatures of up to about 200° C. for as long as necessary to cure the resin.

In general, the textile resin or vinyl monomer with dual functionality when employed may be selected from several general types. According to the materials selected, one of the following processes may be generally followed to achieve the novel garments produced by the present invention. In each type procedure, the methods of application and order of application of the various compounds may be varied as described supra.

TYPE I

1. Apply textile resin and textile resin catalyst to fabric.
2. Dry fabric at temperature that is insufficient to initiate catalysis of the textile resin.
3. Apply polymerizable compound to fabric.
4. Form soil release polymer.
5. Make garment from fabric.
6. Press garment to produce creases where desired.
7. Subject garment to temperature sufficient to catalyze and cure the textile resin.

TYPE II

1. Apply textile resin having more than one type of functional groups, and textile resin catalysts for each functional group to cellulosic fabric.
2. Subject fabric to conditions whereby one type of functional group reacts and remaining functional groups remain dormant.
3. Apply polymerizable compound to fabric.
4. Form soil release polymer.
5. Make garment from fabric.
6. Press creases where desired in garment.
7. Subject fabric to conditions whereby the remaining functional groups are reacted with the cellulose.

TYPE III

1. Apply vinyl monomer having more than one type of functional groups, one type being sites of ethylenic unsaturation and a textile resin catalyst to a cellulosic fabric.

2. Dry the fabric at temperatures such that the textile resin catalyst remains dormant.

3. Apply polymerizable compound to fabric.

4. Subject the fabric to irradiation to form soil release polymer and attach the ethylenic group in the vinyl monomer to the cellulose.

5. Make a garment from the fabric.

6. Produce desired creases in the garment.

7. Subject the garment to textile resin curing conditions.

In each of the above types of procedures, the ultimate curing of the textile resin may be accomplished prior to the manufacture of the garment whereby a good wash-and-wear fabric having soil release properties is produced.

Procedures of Types I, II and III, as is evident, relate to the process of the present invention being applied to a textile material to afford said textile material soil release and durable press or wash-and-wear characteristics. Otherwise than above shown, the polymerizable compound, textile resin catalyst, etc., are just applied to the desired substrate and dried, subjected to textile resin curing conditions, etc., according to the specifications described herein.

The drying temperatures that are insufficient to initiate the catalysis are, of course, dependent upon the particular catalyst being employed. In general, however, the drying step is conducted at a rate of approximately 10 to 70 yards per minute at temperatures ranging from about 225° to 300° F. preferably in a tenter frame. The drying temperature range overlaps to some degree with the curing temperature range set forth below. When drying in the overlapping portion of the drying and curing ranges, it is important that there be no premature curing of the textile resin. Time is the prime variable and when drying the substrate in the higher end of the drying temperature range, care must be taken to avoid heating the substrate for a time sufficient to initiate catalysis that would at least partially cure the textile resin.

Irradiation techniques may be employed according to the process of the present invention to polymerize the polymerizable compound and form the soil release polymer in situ on the textile material. An insulating core transformer, operated at potential varying between one hundred thousand electron volts and five hundred thousand electron volts may be successfully used to irradiate the textile material. Such a transformer is commercially available from High Voltage Engineering Corporation, Burlington, Massachusetts. The amount of ionizing irradiation necessary according to the present invention is at least 32 electron volts for each ion pair formed. Both high energy particle and ionizing irradiation are useful according to the present invention. The preferred dosage of irradiation according to the present invention is in the range of one thousand rads to one hundred megarads, a rad being the amount of high energy irradiation of the type which results in energy absorption of one hundred ergs per gram of absorbing material. More preferably, however, the irradiation dosage ranges from 0.5 to 5 megarads.

Curing is accomplished by subjecting the textile material to conditions such that a crosslinking or insolubilizing reaction occurs. For example, a crosslinking reaction between functional groups of a textile resin and hydroxyl groups of the cellulose in the textile material converts the resin to the thermoset state. Curing the in situ formed acid polymer converts it to an insolubilized state. Temperature is the prime mover and generally a temperature in the range of 100° C. to about 300° C. is sufficient. The curing medium that supports the necessary temperature may be any substance that is inert to both the fabric and the ingredients applied thereto, e.g. hot air, steam, etc. In the instance with a vinyl monomer which possesses two different types of functional groups, there are actually two reaction steps, the first being conducted at a temperature lower than the second and insufficient to initiate the second reaction, e.g., a first graft polymerization step in which the vinyl monomer becomes chemically attached to the cellulose, and a subsequent curing step in which the remaining functional groups on the grafted polymer react with the hydroxyl groups of the cellulose forming crosslinks.

The duration of the various processing steps varies diversely with the particular ingredients employed. In each situation, however, the treatment time is that necessary to sufficiently cause insolubilization and/or curing preferably about 0.1 to 30 minutes.

The following examples illustrate preferred embodiments of the present invention but are not intended to restrict the scope of the invention. In the examples, parts and percentages are by weight. The fabrics prepared in accordance with the procedures set forth in the examples are tested for soil release according to the following procedures. The soil release values are determined by comparison to a set of standards having numerical ratings from 1.0 to 5.0, with 1.0 representing no stain removal and 5.0 being complete removal of the stain. The fabrics are stained with mineral oil. After staining, the fabric is washed one time in a Kenmore automatic washer using one cup of All detergent (sold by Lever Brothers) and a wash water temperature of about 140° F. The fabric is dried for approximately 40 minutes at a temperature of about 160° F. The stains in the dried fabric are compared with the set of standards. The values listed in the Tables under the headings 5 and 10 washes represent staining after 5 or 10 normal washings and then a single wash to remove the stain.

EXAMPLE I

Seventy parts acrylic acid, 30 parts butyl acrylate and 20 parts water are combined and the formulation applied to a Dacron/cotton (65/35) fabric with an applicator roll. The wet pickup on the fabric is about 25 percent.

After about 1 hour hold time, the fabric is subjected to irradiation with an insulating core transformer manufactured by the High Voltage Engineering Corporation of Burlington, Massachusetts. The fabric is passed through the irradiation equipment in a single layer at a speed of about 16 feet per minute, with a dosage of about 3 megarads. The fabric is then laundered at 140° F. and air dried. The resulting fabric having about 9 percent polymer thereon is stained and laundered according to the procedure set forth above to give the soil release ratings reported in Table I.

EXAMPLE II

The procedure of this example is the same as that of Example I except that about 4.5 percent zinc nitrate (50 percent aqueous solution of $Zn(NO_3)_2 \cdot 6H_2O$) is incorporated in the formulation applied to the fabric, and the treated fabric is cured by heating for about 1½ minutes at a temperature of about 160° C. The soiling release results are reported in Table I.

TABLE I

Example	Soil Release Ratings			
	After 1 Wash	After 5 Washes	After 10 Washes	After 20 Washes
Control	2.5	2.0	2.5	2.5
I	5.0	5.0	4.3	4.3
II	4.5	5.0	5.0	4.5

EXAMPLE III

An emulsion is prepared by combining 300 parts water, 2 parts sulfonated castor oil, 70 parts acrylic acid and 30 parts butyl acrylate. The emulsion is padded onto a Dacron/cotton (65/35) fabric and the fabric irradiated immediately with a dosage of about 2 megarads in the irradiation equipment of Example I. The irradiated fabric is then cured by heating at a temperature of about 170° C. for about 15 minutes. The resulting fabric with about 10 percent polymer is laundered at 140° F. and air dried. The soil release ratings of the fabric are reported in Table II.

EXAMPLE IV

To compare the process of the invention with a process employing a preformed polymer, the procedure of Example III is repeated, except that the emulsion padded onto the fabric contains a 10 percent copolymer emulsion of 70 percent acrylic acid and 30 percent butyl acrylate instead of the acrylic acid and butyl acrylate monomers. The emulsion is extremely viscous and very difficult to apply to the fabric. The treated fabric is dried and cured at 150° C. for 15 minutes, but the irradiation step is omitted since a preformed polymer is used. The resulting fabric containing about 5 percent polymer is of unacceptable stiffness both initially and after repeated laundering. The soil release tests show the results set forth in Table II.

EXAMPLE V

The procedure of this example is the same as that of Example IV except that the treated fabric is air dried, instead of being heated at 150° C. for 15 minutes. The test results are set forth in Table II.

TABLE II

Example	Soil Release Ratings		
	After 1 Wash	After 5 Washes	After 10 Washes
Control	2.5	2.0	2.5
III	4.5	5.0	3.8
IV	4.5	4.5	4.5
V	2.5	2.5	2.5

EXAMPLE VI

Dacron/cotton (65/35) fabric is padded with a formulation prepared by mixing 18 percent N-methylol-acrylamide (60 percent aqueous solution) and 4.5 percent zinc nitrate (50 percent aqueous solution of $Zn(NO_3)_2 \cdot 6H_2O$). The treated fabric is dried by heating at 85° C. for about 2½ minutes and then irradiated by passing it through the irradiation equipment of Example I at a rate of about 48 feet per minute with two passes to provide a dosage of about 2 megarads.

The resulting fabric is laundered at 140° F. and then an emulsion containing 75 parts acrylic acid, 25 parts butyl acrylate, 2 parts sulfonated castor oil and 300 parts water is applied. Immediately thereafter, the treated fabric is irradiated with a 4-megarad dosage in a four-pass treatment. The fabric is then dried, pressed on a hot-head press, using a cycle of 5 seconds steam, 5 seconds bake and 5 seconds vacuum and cured by heating at 305° F. for about 15 minutes.

The resulting fabric which contains about 10 percent polymer is subjected to staining and laundering tests which are reported in Table III.

EXAMPLE VII

To compare the process of the invention in durable press fabrics with a process using preformed polymers, a pad bath solution is prepared by dispersing in water 18 percent N-methylol-acrylamide (60 percent aqueous solution); 4.5 percent zinc nitrate (50 percent aqueous solution of $Zn(NO_3)_2 \cdot 6H_2O$); and 10 percent emulsion copolymer comprising 70 percent ethyl acrylate and 30 percent acrylic acid. The above solution is padded onto samples of Dacron/cotton (65/35) fabric to provide about a 50 percent wet pickup. The fabric is then dried at about 85 percent C. until the moisture regain is reduced to approximately 8 percent.

The fabric is then irradiated with a 2-megarad dosage and pressed on a hot-head press with a cycle of 5 seconds steam, 5

seconds bake and 5 seconds vacuum. The fabric containing about 1.5 percent polymer is cured for about 15 minutes at a temperature of about 305° F. The test results are reported in Table III.

TABLE III

Example	Soil Release Ratings		
	After 1 Wash	After 5 Washes	After 10 Washes
Control*	1.0	1.5	2.0
VI	5.0	5.0	4.8
VII	3.5	3.0	2.5

*with N-methylol acrylamide

EXAMPLE VIII

A 20 percent solution of calcium acrylate is applied to a Dacron/cotton (65/35) fabric and the fabric irradiated with a 4-megarad dosage. The resulting fabric after being laundered at 140° F. shows a soil release of 5.0 after one wash and 3.3 after five washes.

EXAMPLE IX

A Dacron/cotton (65/35) fabric is padded with a solution containing about 24 percent dihydroxy dimethylol ethylene urea (50 percent solution), 2.3 percent zinc nitrate ($Zn(NO_3)_2 \cdot 6H_2O$), 0.25 percent ethoxylated alkyl phenol and the balance water. The treated fabric is dried and cured at about 350° F. for about 90 seconds in a festoon oven. The cured fabric is then washed with water containing 0.1 percent ethoxylated alkyl phenol, acidified with a 0.1 percent aqueous acetic acid solution, rinsed with water and dried.

The resulting fabric is soaked in a 50 percent aqueous acrylic acid solution maintained at a temperature of about 75° C. for 15 minutes. The fabric is squeezed to remove excess liquid and irradiated at a dosage of about 4-megarads. The low molecular weight surface polymer is removed by soaking the irradiated fabric in a 2 percent aqueous sodium bicarbonate solution, and the fabric is then laundered at a temperature of 140° F. The resulting fabric containing about 19 percent polymer shows the soil release characteristics set forth in Table IV.

EXAMPLE X

The procedure of this example is the same as that of Example IX except that the treatment with the pad bath solution and the curing, washing and drying steps associated therewith are omitted. The resulting fabric containing about 42 percent polymer has the soil release characteristics set forth in Table IV.

EXAMPLE XI

The procedure of this example is the same as that of Example IX except that the concentration of acrylic acid in the aqueous solution is 35 percent. The fabric containing about 7.5 percent polymer shows the soil release characteristics set forth in Table IV.

EXAMPLE XII

The procedure of this example is the same as that of Example XI except that the treatment with the pad bath solution and the curing, washing and drying steps associated therewith are omitted. The resulting fabric containing about 26 percent polymer has the soil release characteristics set forth in Table IV.

EXAMPLE XIII

The procedure of this example is the same as that of Example IX except that the aqueous acrylic acid solution is replaced with a solution containing 55 percent acrylic acid, 20 percent butyl acrylate and 25 percent water. Tests on the fabric with 19 percent polymer show the results set forth in Table IV.

EXAMPLE XIV

The procedure of this example is the same as that of Example XIII except that the treatment with the pad bath solution and the curing, washing and drying steps associated therewith are omitted. The fabric containing about 35 percent polymer shows the soil release values set forth in Table IV.

TABLE IV

Example	After 1 Wash	Soil Release Ratings		
		After 5 Washes	After 10 Washes	After 20 Washes
IX	5.0	5.0	4.8	5.0
X	5.0	5.0	5.0	5.0
XI	5.0	5.0	5.0	5.0
XII	5.0	5.0	4.5	5.0
XIII	5.0	5.0	5.0	5.0
XIV	5.0	5.0	5.0	5.0

EXAMPLE XV

A solution containing 75 parts acrylic acid, 25 parts butyl acrylate, 5 parts benzoyl peroxide and 15 parts water is applied to a Dacron/cotton (65/35) fabric with an applicator roll to provide a 25 percent wet pickup. The treated fabric is rolled up and sealed in a polyester film. The sealed roll is heated in an oven at a temperature of about 80° C. for about two hours. After removal from the oven the fabric is washed with water, a 2 percent sodium bicarbonate solution and rinsed with water again. The resulting fabric containing about 7 percent polymer shows good soil release characteristics similar to those of Example I.

EXAMPLE XVI

The procedure of the above examples is repeated, utilizing other fabrics including fabrics containing viscose rayon, Orlon, Acrilan, acetate, polypropylene, etc., fibers with similar improvements in soil release characteristics.

It will be noted that the fabric prepared in Examples I, X, XII and XIV provide outstanding durability of soil release characteristics even without any curing step. This is an important advantage since it materially reduces the processing time required to produce textile material with durable soil release characteristics. In contrast, the application of a preformed polymer in Example V without the curing step results in a complete loss of the soil release polymer in the initial laundering. Example VI illustrates the combination of a durable press polymer with the in situ formed soil release polymer, as contrasted with the conventional durable press polymer-soil release preformed polymer heretofore utilized as illustrated by Example VII. The superiority of the process of the present invention in combination with the textile resin is apparent from the results set forth in Table III.

It is apparent from the above description and examples that the process of the invention in which a soil release polymer is formed in situ on the textile material provides benefits and advantages not attainable with other systems. Moreover, the present invention provides textile material having improved soil release characteristics. Also, the invention provides fabric having soil release characteristics which are much more durable than heretofore possible. Furthermore, the process of the invention provides such benefits and advantages in combination with durable press and/or wash-and-wear characteristics.

Having thus disclosed the invention, what is claimed is:

1. A process for imparting soil release and durable press characteristics to textile material including cellulosic and/or synthetic fibers which comprises:

- a. applying thereto a monomeric cellulosic cross-linking agent characterized by a vinyl group and an N-methylol amide group and a catalyst therefor;
 - b. subjecting the textile material to high energy irradiation;
 - c. heating the treated textile material to a temperature between about 100° C. and 300° C.;
 - d. applying to the textile material an ethylenically unsaturated monomer having at least one carboxylic acid group and another ethylenically unsaturated monomer copolymerizable therewith; and
 - e. subjecting the textile material to high energy irradiation to form a synthetic acid copolymer comprising at least 10 weight percent acid calculated as acrylic acid.
2. The process as defined in claim 1 wherein the polymerizable compound is a monomeric mixture comprising an acrylic ester and an acrylic acid.
3. A process as defined in claim 1 wherein the textile resin catalyst is selected from the group consisting of metal salts and amino salts.
4. The process as defined in claim 1 wherein the polymerizable compound is a monomeric mixture comprising about 10 to 80 parts of an acrylic ester and about 20 to 90 parts of an acrylic acid.
5. The process as defined in claim 1 wherein the polymerizable compound is a monomeric mixture comprising about 10 to 50 parts butyl acrylate and about 50 to 90 parts acrylic acid.
6. The process as defined in claim 1 wherein the polymerizable compound is a monomeric mixture comprising about 20 to 30 parts butyl acrylate and about 70 to 80 parts acrylic acid.
7. The process as defined in claim 1 wherein the monomeric cellulosic cross-linking agent is N-methylol acrylamide.
8. The process as defined in claim 1 wherein the textile material is subjected to an irradiation dosage of about 0.5 to 5 megarads.
9. The process as defined in claim 1 wherein the textile material is a polyester/cotton blend.
10. A process for imparting soil release and durable press characteristics to textile material including cellulosic and/or synthetic fibers which comprises:
- a. applying thereto an aminoplast textile resin and a catalyst therefor;
 - b. applying to the textile material an ethylenically unsaturated monomer having at least one carboxylic acid group and another ethylenically unsaturated monomer copolymerizable therewith;
 - c. subjecting the textile material to high energy irradiation to form a synthetic acid copolymer comprising at least 10 weight percent acid calculated as acrylic acid; and
 - d. heating the treated material to a temperature between about 100° C. and 300° C.
11. A process for imparting soil release and durable press characteristics to textile material including cellulosic and/or synthetic fibers which comprises:
- a. applying thereto a monomeric cellulosic cross-linking agent characterized by a vinyl group and an N-methylol amide group and a catalyst therefor;
 - b. subjecting the textile material to high energy irradiation;
 - c. applying to the textile material an ethylenically unsaturated monomer having at least one carboxylic acid group and another ethylenically unsaturated monomer copolymerizable therewith;
 - d. subjecting the textile material to high energy irradiation to form a synthetic acid copolymer comprising at least 10 weight percent acid calculated as acrylic acid; and
 - e. heating the treated textile material to a temperature between about 100° C. and 300° C.
12. The process as defined in claim 11 wherein after the irradiation step (d) and before the heating step (e) a garment is tailored from the textile material and treated to produce creases therein.

13. A process for imparting soil release and durable press characteristics to textile material comprising a blend of cellulosic and polyester fibers which comprises:

- a. applying thereto a methylolated ethylene urea and a metal salt or amino salt-catalyst therefor;
- b. heating the treated textile material to a temperature between about 100° C. and 300° C.;
- c. applying to the textile material a monomeric mixture comprising about 10 to 80 parts of an acrylic ester and about 20 to 90 parts of an acrylic acid; and
- d. subjecting the textile material to a high energy irradiation dosage of about 0.5 to 5 megarads to form a synthetic

acid copolymer comprising at least 10 weight percent acid calculated as acrylic acid.

14. A textile material with soil release and durable press characteristics produced according to the process of claim 13.

15. A textile material with soil release and durable press characteristics produced according to the process of claim 1.

16. A textile material with soil release and durable press characteristics produced according to the process of claim 10.

17. A textile material with soil release and durable press characteristics produced according to the process of claim 11.

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