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[54] **TREATMENT OF TEXTILE MATERIAL WITH BORIC ACID**
 11 Claims, No Drawings

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 [51] Int. Cl..... D06m 9/00
 [50] Field of Search..... 8/115.5,
 115.7

ABSTRACT: A process for improving the resistance to discoloration during irradiation of a blended textile material of polyamide and cellulosic fibers which comprises applying to said textile material a metal salt or an amino salt catalyst, boric acid and a monomer containing ethylenic unsaturation and a group capable of reacting with hydroxyl groups and irradiating the treated textile material to polymerize said monomer.

TREATMENT OF TEXTILE MATERIAL WITH BORIC ACID

One of the important processes for treating textile material to achieve minimum care properties, that is, wash and wear or durable press characteristics, involves the application of a polymerizable material to the textile material and the irradiation thereof to graft polymerize the monomer. Subsequently, the textile material is cured generally either in fabric or garment form to provide the minimum care properties.

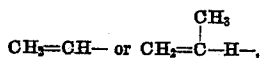
It has been found in such processing of textile material that the irradiation step may cause a change in the color of the textile material. With certain dyed fabrics, there is a tendency for the fabric color to change and with white or undyed fabrics there is a tendency toward yellowing. This condition has limited the available color selections and has increased the quantity of off-quality goods.

In accordance with the present invention, a novel process has been discovered for minimizing the color change of textile material during irradiation. The process of the invention comprises applying a vinyl monomer having dual functionality and boric acid to a textile material and irradiating the treated textile material without causing a significant change in the color thereof. Preferably, the proportion of boric acid based on the dry weight of textile material is between about 0.5 percent and 3 percent and particularly between about 1 percent and 2 percent.

The textile material treated in accordance with the present invention may include any of the conventional natural and synthetic fibers. Advantageously, the textile material includes polyamide fibers in a blend with natural or synthetic fibers and particularly a blend with viscose rayon or cotton fibers.

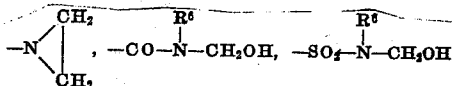
The process of the present invention is particularly useful with polyamide textile material which has been dyed with conventional polyamide acid or dispersed dyestuffs. The color change commonly encountered heretofore was particularly observable with red dyestuffs which tended to lose their color. However, in accordance with the process of the present invention, as pointed out above, such a change is substantially eliminated.

The vinyl monomer with dual functionality employed in the process of the invention advantageously comprises between about 1 percent and 30 percent by weight of the textile material and preferably between about 5 percent and 15 percent. Suitable monomers include compounds containing the group $\text{CHR}^4=\text{CR}^3-$, wherein R^3 is selected from hydrogen and methyl and R^4 is selected from hydrogen and lower alkyl (preferably, containing one to four carbon atoms). The preferred compounds for use in accordance with this invention contain this group, preferably as



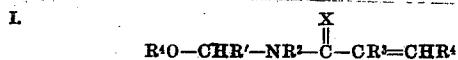
in combination with one of the following reactive groups, preferably adjacent thereto for increased reactivity of the unsaturated group.

Acid reactive groups are generally those found in the textile resins presently employed in the resin treatment of cellulosic fabrics, e.g., methylol, epoxy, acetal, alkylated methylol, aldehyde,



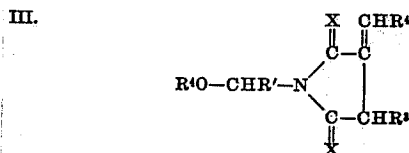
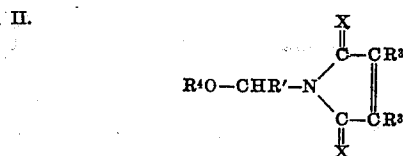
wherein R^6 is hydrogen or alkyl, ---N=C=O , ---N=C=S and the like. These groups are characterized by their ability to combine with the hydroxy groups of the cellulose molecule under textile resin curing conditions.

Typical compounds having both a polymerizable group and a group reactive with hydroxy groups of cellulose include those having the following formulae:

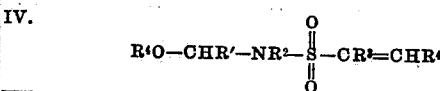


wherein R' is selected from hydrogen, lower alkyl, and the residue of an aldehyde; R^2 is selected from hydrogen, lower alkyl, and $\text{---CX---CR}^3\text{=CHR}^4$; R^3 is selected from hydrogen and methyl; R^4 is selected from hydrogen and lower alkyl; and X is selected from sulfur and oxygen.

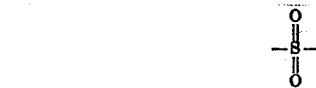
Additional suitable compounds include imides, such as



and the like; vinyl sulfonamides, such as



and the like, wherein R^1 , R^2 , R^3 , R^4 and X are as defined above. The



can have substituted therefor



and sulfonium if desired.

In any of the compounds shown herein, particularly those characterized by formula I above, the

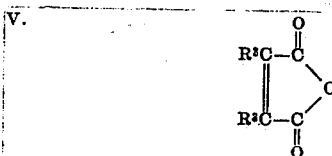


groups may have substituted therefor

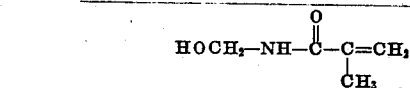


and sulfonium if desired.

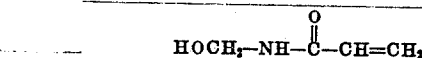
Additional compounds include anhydrides, such as



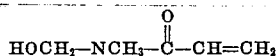
Preferred compounds characterized above are acrylamide compounds such as the methylol acrylamides, e.g.,



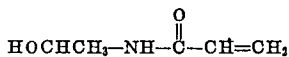
(N-methylol-methacrylamide),



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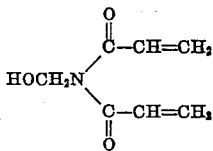


(N-methylol-N-methylacrylamide),



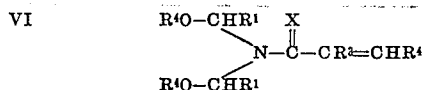
(N-methylmethylolacrylamide),

and



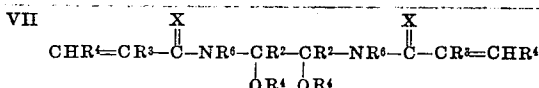
the methylol groups of which are reactive with the hydroxy groups of cellulose under textile resin curing conditions, e.g., from 140° to 200° C., the CH₂ CH—end groups being reactive with polymeric substrates and polymerizable under the influence of irradiation.

Additional suitable acrylamide compounds include those having more than one methylol group, such as shown in the following formulae:

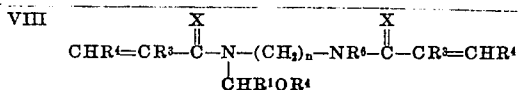


wherein R¹ is selected from hydrogen, lower alkyl and the residue of an aldehyde; R³ is selected from hydrogen and methyl; R⁴ is selected from hydrogen and lower alkyl; and X is selected from oxygen and sulfur.

Other acrylamide compounds may be utilized, e.g., alkylene-bisacrylamides to provide compounds of the following formulae:

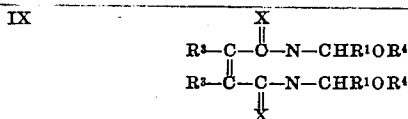


wherein R³, R⁴ and X are as before, R⁶ being hydrogen, lower alkyl or —CHR¹OR⁴ wherein R¹ is as before, at least one R⁶ being —CHR¹OR⁴. Typical compounds include that derived from acrylamide and glyoxal and the N-methylol, di-N-methylol derivatives thereof.



wherein R³, R⁴, R⁶ and X are as before, and n=1-6, e.g., N-methylol-methylene-bis-(acrylamide), methylene-bis-(N-methylol acrylamide) and the like.

Additional, but less preferred, compounds include:



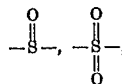
wherein R¹, R³, R⁴ and X are as given above, e.g., as where R¹ and R³ are hydrogen and X is oxygen.

In the above compounds, again in a less preferred embodiment, the



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groups can have substituted therefor



and sulfonium groups.

In preparing the above compounds, aldehydes other than formaldehyde may be utilized, for example, those derived from aldehydes wherein R¹ in the formulae above would be lower alkyl, e.g., acetaldehyde; acetyl, e.g., pyruvaldehyde; OCH(CH₂)_n, wherein n equals 0 to 4, e.g., glyoxal (n=1); OCH(CH₂)₃HOH—, e.g., hydroxy adipaldehyde and the like.

Other compounds having at least one polymerizable group and at least one group reactive with hydroxy groups of cellulose are α-methylol vinyl alkyl ketones, such as α-methylol vinyl methyl ketone; methylol-vinyl-sulfonamides; haloethyl acrylamides and the like.

By the term, "irradiation" as used herein is meant that energy which is propagated through space, the possibility of propagation being unconditioned by the presence of matter, as distinguished from mere mechanical agitation in material such as is characteristic of the energy produced by sonic or ultrasonic transducers, although the speed, direction and amount of energy transferred may be less effective as a result of the presence of matter. The level of irradiation that should be used in accordance with this invention is that having sufficient energy to remove an electronic gas atom forming an ion pair. This required energy is about 32 electron volts for each ion pair formed. Thus, irradiation of at least 32 electron volts is effective for the process of this invention. Both high energy particle irradiation such as electrons, protons, neutrons, α-particles and the like, and ionizing electromagnetic irradiation such as γ-rays, X-rays and the like, are useful and either may be used in accordance with the process of this invention.

Dosages within the range of one thousand to one hundred million rads may be utilized, a rad being the amount of high energy irradiation which leads to an energy absorption of one hundred ergs, in the absorbing material, the preferred range being between 0.1 megarads and 10 megarads, most preferably between 1 and 4 megarads.

The moisture content of the material during irradiation may affect the aesthetic properties thereof. For example, at moisture levels above about 40 percent, a somewhat stiffer product is produced, whereas at moisture levels between about 5 percent and 30 percent, softer products are obtained.

The catalysts useful in activating the 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 acting catalysts are utilized, that is, compounds which are acidic in character under the curing condition. 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, monethanolamine hydrochloride and 2-amino-2-methyl-propanol nitrate. If desired, boric acid may replace a portion or all of the catalyst.

The amount of catalyst to be utilized is that conventionally used in activating the reaction between textile resins and hydroxy groups of cellulose, for example, up to about 5 percent by weight of the catalyst on the fabric with the preferred range being from about 1 percent to about 2 percent. The amount of catalyst to be utilized will depend in part on the temperature at which the reaction is conducted and the amount of catalyst consumed in the reaction.

The curing conditions utilized for garment curing are those conventionally employed in the reaction of cellulosic materials with textile resins. Under these conditions, the dry fabric is heated to an appropriate temperature, for example, from about 100° to 200° C., preferably about 130° to 180° C., to affix the compound to the textile material.

The advantages of the process of the present invention are evident whether the material treated is in the form of fibers,

yarns, threads or fabrics, although the presensitizing embodiment of this invention is most advantageously conducted on fabrics. Such fabrics can be woven, nonwoven, knitted, as desired.

Garments made from these fabrics can be produced in the conventional manner with no additional steps required and 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 up to about 200° C. for as long as necessary to produce the desired results.

The following examples illustrate particular embodiments of the invention but are not intended to limit the scope of the invention. In the examples, parts and percentages are by weight.

EXAMPLE I

A red stretch fabric made from a blend of nylon and viscose rayon fibers (30%/70%) is padded with an aqueous mixture containing about 18% N-methylol acrylamide (60% solution), 5% magnesium chloride catalyst (50% $MgCl_2 \cdot 6H_2O$), 4% Polycryl TF-25 (an acrylic emulsion hand builder), 0.2% nonionic wetting agents and 2% boric acid. The fabric with about 70% wet pickup is dried to about normal moisture regain by heating at about 185° F. and then irradiated with a 1 megarad dose by passing the fabric through irradiation equipment having an insulated core transformer manufactured by High Voltage Equipment Corporation of Burlington, Mass. The fabric is dried in an oven to about normal moisture regain.

A control fabric is made according to the above procedure except the boric acid is omitted from the pad bath and replaced with an equal amount of magnesium chloride catalyst.

Portions of each of the resulting fabrics are made into slacks and pressed on a hot head press with a cycle of 5 seconds steam, 5 seconds bake and 5 seconds vacuum. The slacks are cured in an oven at a temperature of about 320° F. for about 10 minutes. Examination of the finished slacks shows that the slacks treated with the bath containing boric acid have a color substantially the same as the starting fabric while the slacks made with the control fabric have changed shade considerably.

EXAMPLE II

The procedure of this example is the same as that of example I except that the fabric is an olive color and the irradiation dose is reduced to 0.5 megarad. Examination of the slacks shows that the slacks produced according to the invention have a true olive color whereas the slacks made with the control fabric are brown in appearance.

EXAMPLE III

The procedure of this example is the same as that of example I except the irradiation dose is increased to two megarads.

The slacks made according to the process of the invention show the same superiority over the control fabric as observed in example I.

EXAMPLE IV

The procedure of this example is the same as that of example I except an undyed fabric is employed and the fabric is cured in fabric form. The fabric treated in accordance with the invention is without significant yellowing while the control fabric is visibly yellowed.

The above description and examples show that the present invention provides a novel process for minimizing the color change of dyed textile material or yellowing of white or undyed textile material. Furthermore, the invention is especially suitable for the treatment of textile material including polyamide fibers which has been dyed particularly with a red dyestuff.

It will be apparent from the above that various modifications in the formulations and procedures described in detail may be made within the scope of the invention. Therefore, it is intended that the invention be limited only by the following claims.

That which is claimed is:

1. A process for improving the resistance to discoloration during irradiation of a blended textile material of polyamide and cellulosic fibers which comprises applying to said textile material a metal salt or an amino salt catalyst, boric acid and a monomer containing ethylenic unsaturation and a group capable of reacting with hydroxyl groups and irradiating the treated textile material to polymerize said monomer.

2. A process according to claim 1 wherein between about 0.5% and 3% boric acid based on the dry weight of textile material is applied.

3. A process according to claim 1 wherein the treated textile material is irradiated with a dose between about 0.1 and 10 megarads.

4. A process according to claim 1 wherein between about 1% and 30% vinyl monomer based on the dry weight of textile material is employed.

5. A process according to claim 1 wherein the catalyst is selected from the group consisting of zinc nitrate and magnesium chloride.

6. A process according to claim 1 wherein the vinyl monomer is an acrylamide compound.

7. A process according to claim 6 wherein the acrylamide compound is N-methylol acrylamide.

8. A process according to claim 1 wherein the textile material has been dyed with a red dyestuff.

9. A process according to claim 1 wherein the vinyl monomer is an acrylamide compound, a catalyst is applied to the textile material and between about 1% and 2% boric acid based on the dry weight of the textile material is applied.

10. A textile material prepared according to the process of claim 1.

11. A textile material including polyamide fibers prepared according to the process of claim 9.

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