(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 257 800**
**A2**

(12) # EUROPEAN PATENT APPLICATION

(21) Application number: **87306530.4**

(51) Int. Cl.⁴: **D06M 15/256** , D06M 13/34

(22) Date of filing: **23.07.87**

The title of the invention has been amended (Guidelines for Examination in the EPO, A-III, 7.3).

(30) Priority: **29.07.86 US 891271**

(43) Date of publication of application:
**02.03.88 Bulletin 88/09**

(84) Designated Contracting States:
**AT BE CH DE ES FR GB GR IT LI LU NL SE**

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(54) **Method and composition for protecting textile materials.**

(57) Ultra-violet screening compounds have been combined with hydrophobic fluid and solid-repellent compounds to provide a spray, and ultimately a thin solid adherent layering, deposit or coating for fabrics and the like that simultaneously imparts both fade or photodegradiation-resisting properties, and fluid and soil repellent properties.

EP 0 257 800 A2

# Method and Apparatus for Protecting Textile Material

The present invention relates to methods of and materials for protecting fibers and fabrics or textiles and the like from both fading and other deletereous effects of ultra-violet radiation and from the soiling effects of water, oil and other soiling elements.

The art is replete with ultra-violet radiation screening or stabilizing agents for incorporation with synthetic fibers and other articles useable to form fabrics for household furnishings and other purposes and articles of clothing and the like that are to be protected from fading, degradation, deterioration and discoloring by the ultra violet rays. Among such, for example, are U.S. Letters Patent No. 3,888,821 (disclosing the use of a substituted benzotriazole, benzophenone or triazine, for example, absorbed in an aromatic polyamide fiber); 3,379,675 (disclosing benzotriazole and a tris-phenol, for example, for stabilizing polyether-based spandex fibers); and 4,251,433 (disclosing the coating of the extruded organic fibers or other articles with heterocyclic ester ultra-violet stabilizers). The mechanism for such screening action is believed to reside in one or more of filtering action or preferential absorption of deleterious incident wavelengths with dissipation through heat fluorescence or similar phenomena. Sometimes the UV-screening compounds are "spun-in" prior to fiber extrusion, sometimes "dyed in", sometimes "coated-on", and sometimes microdispersed. Similar compounds have also been used for UV-absorption when applied to the human skin (U.S. Letters Patent No. 3,004,896, for example) and to photographic dye images and the like (Nos. 4,447,511 and 4,308,.328, as illustrations).

As an entirely separate and heretofore unrelated art of fabric or textile treatment, numerous different-propertied compounds have been coated on the fabric material, or the same has otherwise been finished therewith, for imparting water, soil, grease or oil repellency, durability against laundering and abrasion and related purposes quite different from UV screening and the phenomena underlying the same. Examples of such finishing compounds are disclosed in, as illustrations, U.S. Letters Patent Nos. 3,549,698; 3,733,357; 3,786,089; 3,949,112; 4,077,770; 4,192,754; 4,219,625; 4,401,780; 4,472,466; 4,473,371; 4,518,649; 4,539,006. Suitable compounds for this very different function include fluorinated polyesters, fluoromethylated diene polymers and copolymers, fluorochemical soil release agents, polyfluoroalkyl compositions and similar compounds.

It has not heretofore been apparent that either there is or can be a relationship between the very differently performing types of compounds used for ultra-violet screening phenomena and those imparting repellent properties by very different phenomena, or that such compounds can be somehow combined or unitized without interfering or chemical interacting or other property-destroying effects, so as to permit the functioning of these distinct screening and repellent phehomena simultaneously; and particularly with a thin enough combined layering or absorption that also maintains the hand, color, strength and other original properties of the fibers or fabric, and that, in such combination, can, where desired, be efficaciously sprayed.

Underlying the present invention is just such a discovery wherein it has surprisingly been found possible to combine ultra-violet screening agents and water, soil and grease-repelling agents as a thin film fabric finish or spray deposition or the like, without impairing the UV or stabilizing or absorbing properties imparted by the former or the efficacy of the repellency properties imparted by the latter and without deleterious chemical interaction or impaired adhesion even though combined together in such thin film.

An object of the invention, accordingly, is to provide a new and improved method of and composite material for simultaneously imparting ultra-violet screening and absorbing properties and fluid and soil repellent properties to organic and related fibers and fabrics or textiles, and without sacrificing the desired hand, coloration, flexibility or other original properties of the same.

A further object is to provide a thin solidified admixed layer or coating imparting such novel properties.

Other and further objects will be explained hereinafter and are more particularly delineated in the appended claims.

In summary, the invention provides a method of simultaneously providing UV screening and fluid and soil repellent properties to organic fibers, fabrics and the like, that comprises, combining a fluid and soil repellent solution with a UV screening compound soluble in and non-reactive with said solution, thoroughly dissolving the said screening compound in the repellent solution in a ratio range of from 5-30% to 95-70% to provide sprayable particles from a few to several hundred microns (say, 1-300), and spraying the same as a thin deposit upon the fabric. Thus the invention provides a novel composition of matter containing an appropriate ultraviolet screening agent combined with a suitable water-repellent coating, formulated

so that the composition can be applied to the surface of a variety of substrates, especially dyed fabrics, to protect them simultaneously from photodegradation and from staining as by various soiling agents, with the composition being applied by spraying or coating, to maximize efficiency. Preferred and best mode steps and materials are hereinafter set forth.

As before stated, underlying the invention is the discovery that a particular combining of ultra-violet screening agents and fluid and soil repellent agents, and the thin film application thereof to preferably organic fiber fabric or textile materials, for example, can result in the simultaneous imparting of both fade-resistant (and other deterioriation) properties and resistance or repellence to wetting or soiling.

As will be more evident from the experimental results delineated in the following examples, preferred compositions comprise from about 2% to 30% of the screening agent (preferably hydroxyl benzotriazoles) in about 98% to 70% of preferably a binder as of the fluorinated type used in 3M's "Scotchguard" trademark product (see, for example, 3M U.S. Letters Patent Nos. 3,981,928 and 4,043,923) or Dupont's polyfluorinated polymers marketed under the trademark "Teflon".

Other screening agents which are useful are hydroxy-benzophenones, e.g., Cyasorb 24, sold by American Cyanamid Co., etc. Other fluorinated binders, as well as binders which lack fluorine, but possess similar functional properties, i.e., hydrophobic character (e.g. polystyrene methyl methacrylate), can also be used. The weight percentage is based on solid binder, which may contain a plasticizer. Optionally, moreover, if desired, the spray composition may contain a fugitive dye which will permit the user to determine which areas of the surface have been sprayed. Examples of useful fugitive dyes are conventional indicator dyes, in combination with a volatile base, e.g., a mixture of phenpohthalein with ammonia, or well-known oxygen-reactive color fading dyes.

## Example 1

An acrylate solution (Rohm & Haas B72) was mixed with Riedel-DeHaen AG film and plastic discoloration UV inhibiter compounds Type HMB ("Riedel" (2-hydroxy-4-methoxybenzophenone) soluble in and non-reactive in the acrylate solution, in the ratio of approximately 4 parts to 98 parts of acrylate solution, and the combination was thoroughly admixed and mutually dispersed. The mixture was reduced with solvent to spraying viscosity. The same was then sprayed from an atomizing spray device in droplets of average size of about 5 microns as a thin layer (of the order of about 20 microns) on dry polyester fabric, and permitted to dry in an adhered thin film form.

The fabric was subjected to ultra-violet rays from a carbon arc lamp for 100 hours with noticeable improvement in fading properties. Water and oil droplets applied to the protected fabric were readily wiped off without strain both before and after the UV tests.

## Example 2

Three grams of o-hydroxyphenylbenzotriazole UV-absorber material ($C_{27}$ $H_{36}$ Cl $O_3$--5-tertiary butyl-3-(5-chloro-2H-benzotriazol-2-yl)-4-hydroxy-benzene-propionic acid octyl ester--Ciba Geigy "Tinuvin 10f", sometimes referred to as T-109) were dissolved, as above, in 50 grams of a fluorinated binder solution containing 12% solids (340 grams solution, 41 grams solids), as marketed under the trademark "Scotchguard", before referenced, to form a sprayable composition of about 5% UV screening agent and 95% repellent solution. The same was used as a spray with fluorinated hydrocarbon (DuPont's "Freon" solvents and "Freon"-ethanol mixtures) producing spray particles of the order of a few microns. Two sprayings upon dyed fabric, from about 6 inches away, were found to apply a thin adherent solid layer or coating that in dried spray particle form (1.2 grams of solid admixed coating consisting of 0.4 grams T-109), producing satisfactory fade stability and stain repellency results similar to those now reported for Example 3.

## Example 3

Another Ciba-Geigy o-hydroxyphenylbenzotriazole UV absorber ("Tinuvin 343") was mixed in the same "Scotch guard" type repellent solution of Example 2 (20% solids) and stirred to get all of the UV-absorber into solution, but in the ratio of 2 grams of T-343 to 50 grams of repellent solution. The solution was placed in a 100 ml bottle attached to a Chromist Spray apparatus (Gelman Instrument Co., Ann Arbor, Michigan), and a fine spray of several micron particle size was directed on dyed fabric (of nylon and cotton and polyester fibers) of red, blue and yellow colors for comparison with (1) unsprayed areas of the fabric and (2) unsprayed fabric areas fronted with an opaque cardboard layer. The same was exposed to a carbon arc generating substantial UV radiation for 160 hours with the following results as determined by fadeometer observations: 8 of 9 of the unsprayed fabric sam-

ples of all three colors showed definite photodegradation or fading effects as compared with the areas covered by the opaque cardboard; while all of the sprayed fabric samples showed either no signs of fading whatsoever or negligible degradation. The sprayed fabric simultaneously maintained its water repellency, as well.

Example 4

A screening agent of o-hydroxybenzophenones ("Cyasorb 24" of American Cyanamid Co.) dissolved in "Scotchguard" fluorinated binder in the ratio of 7 to 93%, and spraydried upon fabric.

Example 5

"Tinuvin" 343 and/or 109 in hydrophobic polystyrene methyl methacrylate repellent solution (in proportions as in Example 2), with a "Freon TA" propellant.

Example 6

The formulation of Example 3 with a fugitive dye indicator (phenophthalein-ammonia) to show the sprayed areas combined with an ammonia volatile base. Alternatively, thymolphthalein indicator may be used that changes from blue to colorless on pH change.

With UV screening elements of the type of the T-109 and T-343, etc. preferred limits of weight per square foot are from about 0.1 gm/ft$^2$ to about 1 gram, if slight yellowing is not desired. For brown or red colored fabrics that do not show the yellowing or shade shift, up to several grams can be used. The preferred limits of the "Scotchguard" type repellent is from a few tenths to about 3 grams/ft$^2$ depending upon the degree of repellency protection desired. The dried spray particle combination layer, indeed, appears to make more effective use of the UV absorber than without the repellency product combined therewith.

Further modifications will occur to those skilled in this art, and such are considered to fall within the spirit and scope of the invention as defined in the appended claims.

## Claims

1. A method of simultaneously providing UV screening and fluid and soil repellent properties to organic fiber fabrics and the like, that comprises, combining a fluid and soil repellent solution with a UV screening compound soluble in and non-reactive with said solution, thoroughly dissolving the said screening compound in the repellent solution in a ratio range of from 2-30% to 98-70% to provide sprayable particles, and spraying the same as a thin deposit upon the fabric.

2. A method as claimed in claim 1 and in which said repellent solution is selected from the group consisting of acrylates and fluoropolymers, and the UV screening agent is selected from the group consisting of o-hydroxybenzophenones and o-hydroxyphenylbenzatriazole.

3. A method as claimed in claim 1 and in which the limits of weight per square foot of spray application are adjusted to from about 0.1 gram/ft$^2$ to about 1 gram/ft$^2$ of screening compound and from a few tenths gram to about 3 grams/ft$^2$ of repellent.

4. A fade-resistant and fluid and soil repellent spray comprising a mixture of a UV screening compound dissolved in a non-reactive repellent solution to permit of spraying of particles thereof of the order of microns.

5. A fade resistant and fluid and soil repellent spray as claimed in claim 4 and in which the ratio range of screening agent and repellent solution is of the order of from about 2-30% to 98-70%.

6. A fade resistant and fluid and soil repellent spray as claimed in claim 5 and in which the limits of weight per square foot of spray are from about 0.1 gram/ft$^2$ to about 1 gram/ft$^2$ of screening compound and from about a few tenths gram to about 3 grams/ft$^2$ of repellent.

7. A fade-resistant and fluid and oil repellent spray as claimed in claim 4 and in which the UV screening agent is selected from the group consisting of acrylates and fluoropolymers and the repellent solution is selected from the group consisting of hydroxybenzophenones and o-hydroxyphenylbenzotriazole.

8. A new composition comprising an ultraviolet screening compound dissolved in a hydrophobic fluid-repellent binder.

9. A new composition as claimed in claim 8 and in which the same is combined with a fluorinated hydrocarbon propellent.

10. A new composition as claimed in claim 8 and in which the screening compound comprises one of hydroxybenzophenone and hydroxyphenylbenzotriazole and the hydrophobic binder comprises one of fluorinated binders and polyester methyl methacrylates.

11. A new composition as claimed in claim 8 and in which the screening compound is from about 0.1 gram/ft$^2$ to about 1 gram/ft$^2$ and the repellent from a few tenths gram/ft$^2$ to about 3 grams/ft$^2$.

12. A new composition as claimed in claim 8 and in which a fugative dye indicator is admixed.

13. A new composition as claimed in claim 9 and in which the screening compound comprises one of "Tinuvin" 109 and 343 and the hydrophobic binder comprises one of "Scotchguard" and "Teflon" binders and the propellent comprises one of "Freon" and "Freon"-methanol mixtures.

14. A fade-resistant and water-repellent solid layer of ultraviolet screening compound intimately admixed with a hydrophobic compound.

15. A layer as claimed in claim 14 of thickness of the order of microns.

16. A layer as claimed in claim 14 and in which the screening compound is one of a hydroxybenzophenones and o-hydroxyphenylbenzotriazole, and the hydrophobic compound is a fluorinated binder.

17. A method of indicating the extent of spray treatment coating on fabrics or the like, that comprises, spraying the fabric or the like with coating surface-treating particles, and including in the spray an admixed fugitive dye indicator to indicate the extent of the sprayed coating during the spraying while thereafter becoming colorless.

18. A method as claimed in claim 17 and in which the surface-treating particles comprise an ultraviolet screening compound dissolved in a hydrophobic fluid-repellant binder.