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# TEXTILE TREATING COMPOSITION

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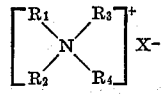
Int. Cl. D06m *13/46;* B44d *5/06*
U.S. Cl. 252—8.75 1 Claim

## ABSTRACT OF THE DISCLOSURE

A textile treating composition essentially consisting of the following three ingredients (1) to (3):

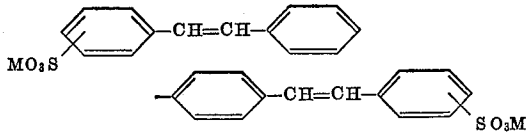
(1) 1 to 10 parts by weight of a cationic surface active agent of the general formula:



wherein $R_1$ and $R_2$ are alkyl radicals of 12–22 carbon atoms, $R_3$ and $R_4$ are alkyl radicals of 1–4 carbon atoms and $X^-$ is a halogen ion, $CH_3SO_4^-$ or

$$C_2H_5SO_4^-,$$

(2) 0.05 to 0.5 part by weight of an optical brightener of the general formula:



wherein M is an alkali metal or an ethanol amine, and

(3) 0.5 to 10 parts by weight of a cationic surface active agent which is characterized by that

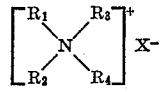
(i) it has one or more of cationic nitrogen atoms in a molecule,

(ii) it has 1 to 3 polyoxyethylene groups attached on said cationic nitrogen atoms, each of the polyoxyethylene groups having a terminal hydroxy radical and having oxyethylene units of 1–14 moles, and

(iii) it has one or two of $RCH_2$-, $RCONH(CH_2)_n$- or $RCOO(CH_2)_n$-, in which R is an alkyl radical of 11–21 carbon atoms and *n* is an integer of 1 to 3, in a molecule.

The present invention relates to a textile treating composition. More particularly, the present invention relates to a fabric softener having optical brighteners incorporated therewith.

Domestic fabric softeners have excellent effects such as softening of textile for providing nice feeling, preventing the accumulation of electrostatic charge on synthetic textile, conditioning textile for smooth ironing and shortening the drying time. Main ingredients of domestic fabric softeners are generally cationic surface active agents of the following general Formula I:



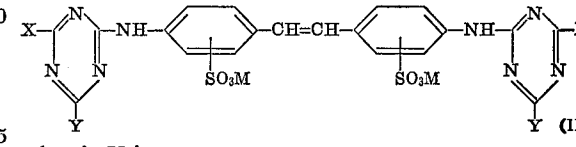
(I)

wherein $R_1$ and $R_2$ represent alkyl radicals of 12–22 carbon atoms, $R_3$ and $R_4$ represent alkyl radicals of 1–4 carbon atoms, and $X^-$ represents a halogen ion, $C_2H_5SO^-_4$ or $CH_3SO_4^-$.

Although domestic fabric softeners have such various excellent effects as mentioned above, they have a disadvantage that, when they are applied to commercial textiles which are finished with an anionic optical brightener, the textiles are often yellowed, because the anionic optical brightener reacts with the cationic softening agent and then fluorescence is quenched. In order to prevent the textiles from yellowing, optical brighteners are usually added to softening agents. However, when any of usual softening agents is employed, the quantity of the optical brightener adsorbed on the textile is rather small so that a sufficient whitening effect is never expected.

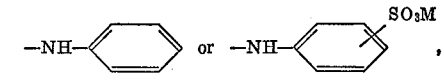
Some studies have been published which were made for the purpose of overcoming these defects, which are as follows:

(A) Optical brighteners of bis-(triazinylamino) stilbenedisulfonicacid having the following general Formula II, which is compatible with said cationic surface active agent represented by the above general Formula I have been described in "Optical Brighteners in Fabric Softeners" [Soap & Chemical Specialities, 40, 85–88 (May in 1965)].

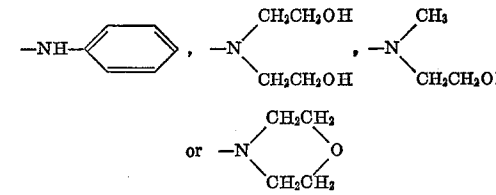


(II)

wherein X is



Y is



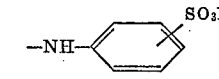
and M represents Na, K or an ethanolamine.

When these optical brighteners are employed, the compositions have to satisfy the following two requirements, that is;

(a) They must contain no inorganic electrolyte.

(b) The solubility of the complex salt of the cationic surface active agent and the optical brightener in water should be large.

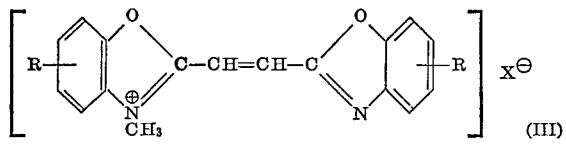
However, in order to satisfy the above requirement (b), X of the optical brightener represented by the above general Formula II should be



and Y should be a radical of the structure having remarkably hydrophilic hydroxyethyl groups, and when such a requirement is chosen, the composition is surely stable. However, as described in the papers and shown in the hereinafter described examples, when this composition is employed for the treatment of textiles, the adsorbed optical brightener is scarcely detected, and, therefore, it cannot be employed practically. It is impossible to prevent the quenching of fluorescence on textiles which is caused by a cationic surface active agent, and to bring about an improvement of the substantivity of optical brighteners incorporated in a fabric softener.

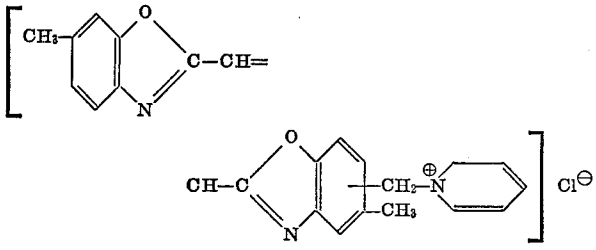
(B) Since a cationic surface active agent such as the compounds as shown by the Formula I is used as a fabric softener, it is expected that, if a cationic optical brightener is added to a fabric brightener, the substantivity of the optical brightener might be improved. As such cationic

optical brightener, there are, for example, Tinopal AN of the oxycyanine type (produced by J. R. Geigy, Switzerland, which has the following Formula III;



(III)

wherein R is a lower alkyl radical, $X^-$ is an anion such as a halogen ion, etc., and Daitophor AN of the bis-oxazol types (produced by Daito Chemical Co., Japan, which has the following Formula IV;



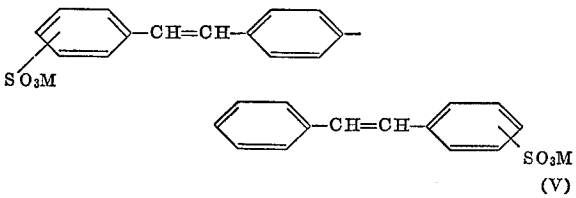
(IV)

In principle, these agents have to show a sufficient whitening effect in a fabric softener, but they have, in fact, an excellent whitening effect only as to poly-acrylonitrile textiles and they do not show a sufficient substantivity to other textiles. Accordingly, they are not desirable optical brighteners in case these agents should be incorporated into the softening agents for underwears, diaper and sweater, etc., which are made of cellulose fibers. Further, most optical brighteners, which are to be blended in detergents and to be used for whitening cellulose textiles, are of anionic stilbene type, so that these anionic optical brighteners react with the aforesaid cationic optical brighteners in a treating bath to form a complex which will cause to decrease the effect of preventing yellowing and the whitening effect. This will be distinctly observed in treating a textile, which has been finished with an anionic optical brightener, by a cycle which is arranged in the order of washing→softening→drying→washing→ . . . . Consequently, it cannot be said that the effects of preventing the yellowing and whitening of textiles by treating textiles with a fabric softener, mixed with a known optical brightener, are satisfactory.

We have studied about the effective prevention of yellowing and the effective whitening of the textile by the use of a fabric softener of which the main ingredient is a cationic surface active agent of the above general Formula I and could attain the purpose by the following novel textile treating compositions.

The composition of the present invention consists essentially of the following three ingredients.

They are a cationic surface active agent of the above general Formula I (first ingredient), sodium distilbenedisulfonate of the following Formula V (second ingredient);



(V)

wherein M represents an alkali metal such as Na or K, or an ethanolamine, and a cationic surface active agent (third ingredient), which is characterized by the facts that (1) it has one or more of cationic nitrogen atoms in a molecule, (2) it has 1 to 3 polyoxyethylene groups attached on said cationic nitrogen atoms, each polyoxyethylene group having a terminal hydroxy radical and



having oxyethylene units of 1–14 moles and (3) it has one or two of

$RCH_2$—, $RCONH(CH_2)_n$— or $RCOO(CH_2)_n$—

(wherein R is an alkyl radical of 11–21 carbon atoms and $n$ is an integer of 1 to 3) in a molecule.

The fabric softener composition consisting essentially of the above three ingredients according to the present invention is excellent in the prevention of yellowing and the whitening of textiles. This composition doesn't react with any of stilbene-type optical brighteners which have been often used for commercially available clothes, so that the yellowing of textiles does not substantially happen. Moreover, the substantivity of the above second ingredient to textiles is better than any known optical brightener. However, in case the fabric softener composition consists only of the first and second ingredients, not containing the third ingredient, the aforesaid effects would not be obtained. In other words, the effect of preventing the yellowing and increasing the whitening effects would not be substantially obtained. The same can be said of a fabric softener composition consisting only of the first and third ingredients, and not containing the second ingredient.

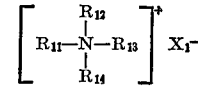
That is to say, the composition of the present invention is not significantly characterized by either one of the second ingredient and the third ingredient, but rather requires the combination of the above first, second and third ingredients. The respective ingredient cannot be replaced by other similar compounds. Effective prevention of yellowing and effective whitening can be obtained only by this combination of the above three ingredients.

In addition, the third ingredient can prevent the "Tinting phenomenon" of the optical brightener. (The "Tinting phenomenon" is a phenomenon that the optical brightener is put in spots on textiles.) Therefore, the composition shows a uniform whitening effect on textile fabrics. Furthermore, the third ingredient reduces the solution viscosity of the fabric softener so that it can be smoothly poured and prevents precipitation after a long storage.

A theoretical explanation of the effects to be obtained by the ternary system of the present invention, which are the prevention of yellowing, the improvement of whitening and the prevention of tinting phenomenon, is not yet possible, but it is presumed to be as follows:

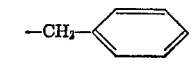
The above cationic surface active agent of the general Formula I and the above optical brightener of the general Formula V are reacted in an aqueous solution to form a complex which tends to be precipitated. However, when a compound having polyoxyethylene groups in the molecule such as the above third ingredient coexists, the complex may stay stably in the solution and the physico-chemical affinity of the complex mainly to cellulosic textiles may be promoted. To this end, it is required that the oxyethylene units in the polyoxyethylene group is about 1 to 14 moles. In case this is more than 15 moles, the nonionic property of the third ingredient will be increased and, consequently, the substantivity thereof will become worse. Moreover, the third ingredient is effective in maintaining the complex in a fine dispersion so that the tinting phenomenon could be prevented.

As the third ingredient of the present invention, any of the above-mentioned cationic surface active agents having the aforesaid structure have these effects. However, the compounds having the following general Formulae VI to XI are preferable.



(VI)

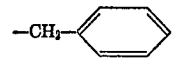
wherein $R_{11}$ and $R_{12}$ represent $-CH_3$, $-C_2H_5$,



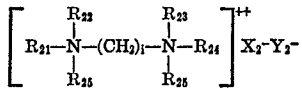
$-CH_2R_{15}$, $-(CH_2)_kNHCOR_{15}$ or $-(CH_2)_kOOCR_{15}$ (wherein $R_{15}$ is an alkyl radical of 11–21 carbon atoms, $k$ is an integer of from 1 to 3), and either one of $R_{11}$ and $R_{12}$ is $-CH_2R_{15}$, $-(CH_2)_kNHCOR_{15}$ or

$$-(CH_2)_kOOCR_{15}$$

$R_{13}$ represents $-CH_3$, $-C_2H_5$,



or $-(CH_2CH_2O)_mH$, $R_{14}$ represents $-(CH_2CH_2)_nH$. $m$ and $n$ each represent an integer of more than 1, and $2 \leqq m+n \leqq 14$, $X_1^-$ represents a halogen ion, $CH_3SO_4^-$ or $C_2H_5SO_4^-$.

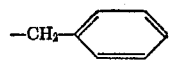


(VII)

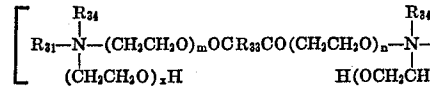
wherein $R_{21}$ represents $-CH_2R_{26}$, $-(CH_2)_kNHCOR_{26}$ or $-(CH_2)_kOOCH_{26}$ (wherein $R_{26}$ is an alkyl radical of 11–21 carbon atoms, $k$ is an integer of from 1 to 3), $R_{22}$, $R_{23}$ and $R_{24}$ represent $-(CH_2CH_2O)_lH$,

$$-(CH_2CH_2O)_mH$$

or $-(CH_2CH_2O)_nH$ respectively, $l$, $m$ and $n$ represent an integer of more than 1 and $3 \leqq l+m+n \leqq 14$, respectively. $R_{25}$ represents $-CH_3$, $-C_2H_5$ or

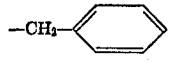


$X_2^-$, $Y_2^-$ represent a halogen ion, $CH_3SO_4^-$ or $C_2H_5SO_4^-$, and $i$ represents an integer of from 2 to 3.



(VIII)

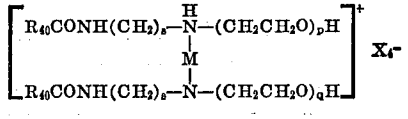
wherein $R_{31}$ and $R_{32}$ represent alkyl radicals of 12–21 carbon atoms, $R_{33}$ represents an alkylene radical of more than 2 carbon atoms, $R_{34}$ represents $-CH_3$, $-C_2H_5$ or



$m$ and $n$ represent an integer of more than 1 and

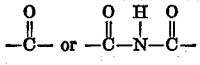
$$2 \leqq m+n \leqq 14$$

respectively, $x$ and $y$ each represent an integer of more than 1 and $2 \leqq x+y \leqq 14$, respectively, $X_3^-$, $Y_3^-$ represent a halogen ion, $CH_3SO_4^-$ or $C_2H_5SO_4^-$.

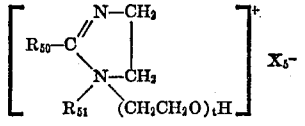


(IX)

wherein M represents



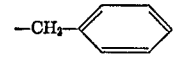
$R_{40}$ is an alkyl radical of 12–21 carbon atoms, $s$ is 2 or 3, $p$. $q$ are an integer of more than 1 and $2 \leqq p+q \leqq 14$, respectively, $X_4^-$ is an anion which will be derived from acetic acid, lactic acid, hydroxyacetic acid, boric acid or benzoic acid by removing one hydrogen from these acids.

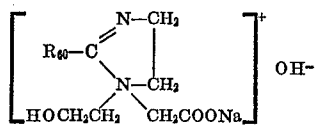


(X)



wherein $R_{50}$ is an alkyl radical of 11–21 carbon atoms, $X_5^-$ is a halogen ion, $CH_3SO_4^-$ or $C_2H_5SO_4^-$, $t$ is an integer from 1 to 14, $R_{15}$ is $-CH_3$, $-C_2H_5$ or





(XI)

wherein $R_{60}$ is an alkyl radical of 11–2- carbon atoms.

As described above, the composition according to the present invention consists essentially of a combination of the above three ingredients and its unexpected remarkable effects could be obtained by the synergistic action of these ingredients.

The purpose of the present invention can be attained by blending said three ingredients in an appropriate blending ratio. It is preferable to blend these ingredients in the ratio that the first ingredient is 1 to 10 parts, the second ingredient is 0.05 to 0.5 part and the third ingredient is 0.5 to 10 parts respectively, in which all parts are parts by weight.

The present invention is further illustrated by the following examples.

## EXAMPLE

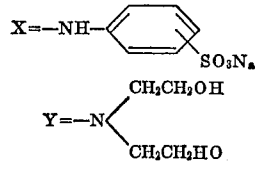
The details of the three ingredients which were used in the following formulations according to the present invention are as follows:

Softening agent (first ingredient)

| | Percent by wt. |
|---|---|
| No. 1: | |
| Distearyl dimethyl ammonium chloride | 75 |
| Isopropylalcohol | 19 |
| Water | 6 |
| No. 2: | |
| Dilauryl diethyl ammonium ethylsulfate | 75 |
| Isopropylalcohol | 19 |
| Water | 6 |

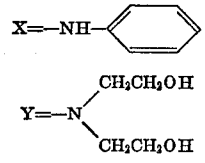
Optical brightener (second ingredient)

No. 1: An optical brightener having the above Formula II, in which



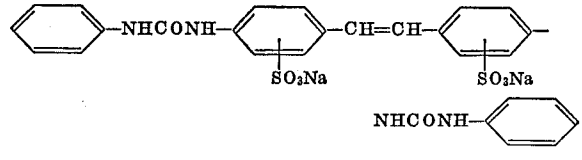
and which is commercially available under the trade name of Tinopal 2B (produced by J. R. Geigy). Its $\lambda_{max}$ is 440 m$\mu$.

No. 2: An optical brightener having the Formula II, in which



Its $\lambda_{max}$ is 440 m$\mu$.

No. 3: An optical brightener having the formula



which is commercially available under the trade name of Whitex R Extra Conc. (produced by Sumitomo Chemical Co.). Its $\lambda_{max}$ is 425 m$\mu$.

No. 4: An optical brightener of the general Formula III, which is commercially available under the trade name of Tinopal AN (produced by J. R. Geigy). Its $\lambda_{max}$ is 420 m$\mu$.

No. 5: An optical brightener of the general Formula IV, which is available under the trade name of Daitophor AN (produced by Daito Chemical Co.). Its $\lambda_{max}$ is 430 m$\mu$.

No. 6: An optical brightener of the general Formula V. Its $\lambda_{max}$ is 435 m$\mu$.

Before the use, optical brighteners were purified by precipitating crystals through acidification and reneutralizing the extracted crystals or by recrystallizing them from ethanol or acetone and then desalting the crystals.

Third ingredient

No. 1: Stearyl-trimethylammonium chloride.

No. 2: Stearyl-di(polyethoxy)methylammonium chloride containing 2 moles of ethylene oxide.

No. 3: Lauryl-di(polyethoxy)ethylammonium ethylsulfate containing 10 moles of ethylene oxide.

No. 4: Myristyl-di(polyethoxy)methylammonium chloride containing 14 moles of ethylene oxide.

No. 5: Stearyl-di(polyethoxy)methylammonium chloride containing 20 moles of ethylene oxide.

No. 6: Stearyl-polyethoxymethylbenzylammonium chloride containing 8 moles of ethylene oxide.

No. 7: Stearyl-polyethoxymethylbenzylammonium chloride containing 20 moles of ethylene oxide.

No. 8: Dicoconutalkyl-polyethoxymethylammonium chloride containing 2 moles of ethylene oxide.

No. 9: N-stearyl-N,N'-diethyl-tri-(polyoxyethylene) ethylene-diammonium diethylsulfate containing 10 moles of ethylene oxide.

No. 10: N-lauryl-N,N'-dimethyl-tri(polyoxyethylene) propylene-diammonium dichloride containing 3 moles of ethylene oxide.

No. 11: N-stearoylaminoethyl-N,N-dimethyl-tri-(polyoxyethylene)ethylene-diammonium dichloride containing 12 moles of ethylene oxide.

No. 12: N-lauroyloxypropyl-N,N-diethyl-tri-(polyoxyethylene)-propylene-diammonium ethylsulfate containing 10 moles of ethylene oxide.

No. 13: N-lauryl-N,N'-dimethyl-tri-(polyoxyethylene) propylenediammonium dimethylsulfate containing 30 moles of ethylene oxide.

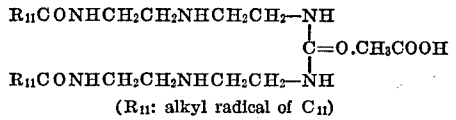
No. 14: A compound shown by the general Formula VIII, wherein $R_{31}$, $R_{32}$ are aluryl radicals, $R_{33}$ is an adipic acid residue of 4 carbon atoms, $R_{34}$ is methyl, $m+n=10$, $x+y=10$ and $X_3^-, Y_3^- = Cl^-, Cl^-$.

No. 15: A compound which is same with the compound No. 14, except that $m+n=20$ and $x+y=20$.

No. 16: A salt of N,N'-distearoylaminoethyl-N,N'-dihydroxyethylurea with acetic acid (the mole ratio is 1:1).

No. 17: N,N'-dilauroylaminoethyl-N,N'-di(polyoxyethylene)biuret lactate containing 10 moles of ethylene oxide.

No. 18: N,N'-dilauroylaminoethyleneaminoethyleneurea acetate:

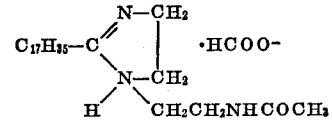



($R_{11}$: alkyl radical of $C_{11}$)

No. 19: 1-ethyl-1-hydroxyethyl-2-stearylimidazolinium ethylsulfate of the Formula X.

No. 20: Sodium 1-hydroxyethyl-1-carboxy-2-stearylimidazoliniumhydroxide of the Formula XI.

No. 21: 1-(acetylaminoethyl)-2-heptadecyl imidazolinium formate of the formula:




No. 22: Polyoxyethylene nonoylphenylether ($\overline{P}=10$).

Formulation 1:

| | Parts by wt. |
|---|---|
| Softening agent No. 1 | 8 |
| Optical brightener No. 6 | 0.2 |
| The third ingredient No. 1 | 2 |
| Ethylene glycol | 6 |
| NaCl | 0.05 |
| Water | Balance |

The total amount is made to be 100 parts by weight in all formulations.

Formulation 2:

As the third ingredient No. 2 was used and other materials were same as in Formulation 1.

Formulation 3:

| | Parts by wt. |
|---|---|
| Softening agent No. 1 | 9 |
| Optical brightener No. 6 | 0.2 |
| The third ingredient No. 3 | 1.5 |
| Ethylene glycol | 6 |
| KCl | 0.02 |
| Water | Balance |

Formulation 4:

Except using No. 4 as the third ingredient, other materials were same as in Formulation 3.

Formulation 5:

Except using No. 5 as the third ingredient, other materials were same as in Formulation 3.

Formulation 6:

| | Parts by wt. |
|---|---|
| Softening agent No. 2 | 8.0 |
| Optical brightener No. 6 | 0.3 |
| The third ingredient No. 6 | 3 |
| Diethylene glycol | 5 |
| $NH_4Cl$ | 0.04 |
| Water | Balance |

Formulation 7:

Except using No. 7 as the third ingredient, other materials were the same as in Formulation 6.

Formulation 8:

Except using No. 8 as the third ingredient, other materials were same as in Formulation 6.

Formulation 9:

| | Parts by wt. |
|---|---|
| Softening agent No. 1 | 5 |
| Optical brightener No. 6 | 0.1 |
| The third ingredient No. 9 | 5 |
| Isopropylalcohol | 5 |
| NaCl | 0.05 |
| Water | Balance |

Formulation 10:

Except using No. 10 as the third ingredient, other materials were same as in Formulation 9.

Formulation 11:

Except using No. 11 as the third ingredient, other materials were same as in Formulation 9.

Formulation 12:

Except using No. 12 as the third ingredient, other materials were same as in Formulation 9.

Formulation 13:

Except using No. 13 as the third ingredient, other materials were same as in Formulation 9.

Formulation 14:

| | Parts by wt. |
|---|---|
| Softening agent No. 1 | 7 |
| Optical brightener No. 6 | 0.2 |
| The third ingredient No. 14 | 3 |
| Ethanol | 5 |
| Urea | 3 |
| NaCl | 0.05 |
| Water | Balance |

Formulation 15:
Except using No. 15 as the third ingredient, other materials were same as in Formulation 14.

Formulation 16:

| | Parts by wt. |
|---|---|
| Softening agent No. 1 | 6 |
| Optical brightener No. 6 | 0.4 |
| The third ingredient No. 16 | 4 |
| Propylene glycol | 5 |
| NaCl | 0.02 |
| Water | Balance |

Formulation 17:
Except using No. 17 as the third ingredient, other materials were same as in Formulation 16.

Formulation 18:
Except using No. 18 as the third ingredient, other materials were same as in Formulation 16.

Formulation 19:

| | Parts by wt. |
|---|---|
| Softening agent No. 2 | 5 |
| Optical brightener No. 6 | 0.2 |
| The third ingredient No. 19 | 5 |
| Ethylene glycol | 2 |
| KCl | 0.05 |
| Water | Balance |

Formulation 20:
Except using No. 20 as the third ingredient, other materials were same as in Formulation 19.

Formulation 21:
Except using No. 21 as the third ingredient, other materials were same as in Formulation 19.

Formulation 22:
Except using No. 22 as the third ingredient, other materials were same as in Formulation 19.

Formulation 23:

| | Parts by wt. |
|---|---|
| Softening agent No. 1 | 8 |
| Optical brightener | 0.2 |
| The third ingredient | None |
| Ethylene glycol | 6 |
| NaCl | 0.05 |
| Water | Balance |

Formulation 24:
Except using No. 1 as the optical brightener, other materials were same as in Formulation 1.

Formulation 25:
Except using No. 3 as the optical brightener, other materials were same as in Formulation 1.

Formulation 26:
Except using No. 2 as the optical brightener, other materials were same as in Formulation 6.

Formulation 27:
Except using No. 4 as the optical brightener, other materials were same as in Formulation 1.

Formulation 28:
Except using No. 5 as the optical brightener, other materials were same as in Formulation 6.

Formulation 29:

| | Parts by wt. |
|---|---|
| Softening agent No. 1 | 8 |
| Optical brightener No. 1 | 0.3 |
| The third ingredient No. 22 | 1.0 |
| Ethylene glycol | 5 |
| NaCl | 0.05 |
| Water | Balance |

Formulation 30:
Except using No. 4 as the optical brightener, other materials were same as in Formulation 29.

With respect to the softener composition of the above formulations, the following tests were conducted.

TEST 1

Judgments of effects concerning the prevention of yellowing the improvement of whitening and the tinting phenomenon

Bleached Cotton Broad #60 swatches (Reflectance: 89) and these broad swatches treated by a stilbene type optical brightener, Whitex BB conc. (produced by Sumitomo Chemical and Industrial Co.—commercial name—$\lambda_{max.}$: 440 m$\mu$) were treated with said softener compositions (Formulations 1 to 30) under the following conditions.

| | |
|---|---|
| Concentration of the softener composition, percent | 0.2 |
| The ratio of the swatch to the aqueous treating solution | 1:100 |
| Treating time minutes | 5 |
| Temperature ° C | 30 |

Sun-dried after removing water centrifugally.

As to the thus treated textiles the reflectance of each swatch was measured by the use of a spectrophotometer (Hitachi fluorophotoreflectometer FR-1 in the EPU-2A type spectrophotometer).

Reflectances of the swatches were measured at the maximum reflection wave $\lambda_{max}$ by using a filter of the effective wave width of 5.0 m$\mu$. These reflectance data do not necessarily show a complete agreement with the naked eye observation.

Therefore, the whiteness was evaluated as the next criteria by 5 persons with their naked eyes under day light, and it was graded by the total of the points evaluated by 5 persons.

Criteria of judgment

| | |
|---|---|
| Sufficiently white in comparison with blank | +2 |
| Fairly white in comparison with blank | +1 |
| No difference | 0 |
| Fairly less white than blank | −1 |
| Remarkably less white than blank | −2 |

The tinting phenomenon was evaluated by examining the treated swatches under ultraviolet light (black light), in which the following criteria were used.

Criteria of judgment

| | |
|---|---|
| No tinting, having the optical brightener uniformly adsorbed | +1 |
| Middle | +3 |
| Much tinting (Formulation No. 25 was presumed to be point 5) | +5 |

The results were shown in Table 1.

TEST 2

The effect of whitening by the repeated treatments

The swatches of Broad #60 (polyester-cotton (35–65) mixed, which was not treated with optical brightener, was washed with a commercial detergent (containing a stilbene-type optical brightener) and the swatches were treated with 0.1% aqueous solution of the above softening compositions in the 1/40 of swatches/solution weight ratio, and squeezed and air-dried. After the washing-softening-drying cycles were repeated 5 and 10 times, the whiteness (reflectance) was examined in the manner as described above.

The results were shown in Table 1.

TEST 3

Stability of the formulations

After storing the aforesaid compositions at −5° C. 20° C., or 40° C. for two months, their stabilities were first examined with eye to check their phase separation or syneresises, etc. and also they were checked under black light as to if they had troubles such as separation of optical brightener or its precipitation, etc.

The results were shown in Table 1.

TABLE 1

Results in the Tests 1 to 3 as to prevention of yellowing, whitening effects, tinting phenomena and solution stability

| Formulation | Prevention effect of yellowing and whitening effect in Test 1: Swatches not finished with optical brightener — Reflectance | Swatches not finished with optical brightener — Judgment with eye | Swatches finished optical brightener — Reflectance | Swatches finished optical brightener — Judgment with eye | Judged value of tinting phenomena | Reflectance by Test 2 — 5 cycles | Reflectance by Test 2 — 10 cycles | Solution stability by Test 3 |
|---|---|---|---|---|---|---|---|---|
| 1 | 92 | 2 | 120 | −3 | 5 | 118 | 119 | Phase separation, precipitation of optical brighteners. |
| 2 | 109 | 9 | 127 | 3 | 1 | 131 | 134 | Stable, bright in appearance. |
| 3 | 111 | 10 | 131 | 6 | 1 | 133 | 136 | Do. |
| 4 | 102 | 7 | 127 | 4 | 1 | 129 | 130 | Do. |
| 5 | 93 | 1 | 117 | −6 | 2 | 130 | 119 | Do. |
| 6 | 111 | 10 | 128 | 5 | 1 | 134 | 136 | Do. |
| 7 | 91 | 1 | 119 | −4 | 1 | 117 | 116 | Do. |
| 8 | 108 | 9 | 126 | 2 | 1 | 133 | 135 | Do. |
| 9 | 112 | 10 | 130 | 7 | 1 | 132 | 134 | Do. |
| 10 | 107 | 10 | 129 | 7 | 1 | 132 | 134 | Do. |
| 11 | 108 | 9 | 129 | 7 | 1 | 134 | 135 | Do. |
| 12 | 111 | 10 | 133 | 8 | 1 | 133 | 136 | Do. |
| 13 | 90 | 0 | 116 | −7 | 1 | 117 | 117 | Do. |
| 14 | 107 | 10 | 128 | 5 | 1 | 133 | 136 | Do. |
| 15 | 89 | −1 | 116 | −7 | 1 | 115 | 114 | Do. |
| 16 | 112 | 10 | 129 | 7 | 1 | 134 | 137 | Do. |
| 17 | 111 | 10 | 129 | 8 | 1 | 133 | 136 | Do. |
| 18 | 95 | 3 | 115 | −7 | 5 | 121 | 119 | Phase separation, precipitation of optical brightener. |
| 19 | 111 | 10 | 127 | 5 | 1 | 134 | 134 | Stable, bright in appearance. |
| 20 | 111 | 9 | 133 | 9 | 1 | 134 | 137 | Do. |
| 21 | 89 | 0 | 115 | −8 | 5 | 115 | 114 | Phase separation, precipitation of optical brighteners. |
| 22 | 91 | 1 | 116 | −7 | 2 | 111 | 110 | Do. |
| 23 | 92 | 2 | 118 | −4 | 5 | 117 | 118 | Do. |
| 24 | 89 | 1 | 114 | −8 | 3 | 121 | 121 | Do. |
| 25 | 90 | 1 | 109 | −4 | 4 | 112 | 112 | Do. |
| 26 | 93 | 2 | 109 | −4 | 3 | 120 | 120 | Do. |
| 27 | 92 | 0 | 107 | −6 | 5 | 109 | 110 | Stable, bright in appearance. |
| 28 | 94 | 2 | 109 | −4 | 5 | 110 | 112 | Do. |
| 29 | 91 | 0 | 119 | −3 | 3 | 121 | 122 | Phase separation, precipitation of optical brightners. |
| 30 | 92 | 0 | 112 | −1 | 3 | 110 | 111 | Stable, bright in appearance. |
| Blank | 89 | 0 | 124 | 0 | ---------- | 125 | 128 | |

The following remarks are made on the results shown in Table 1. First, the basis of the interpretation of the data is follows: The reflectance of the swatch not treated with optical brightener is 89 at $\lambda_{max}$ 440 mμ and, therefore, values over 89% prove whitening effects. The reflectance of the swatch treated with optical brightener (Whitex BB conc.) is 124 and, therefore, the swatches under 124 prove that they are quenched or yellowed by the cationic softening agent. On the other hand, the swatches over 124 prove that they are whitened by the fabric softener.

The same standard of evaluation will apply to the values of the swatches treated 5 and 10 times in the Test 2.

Now, the remarks are as follows:

(1) The formulations 1 to 8 are examples in which the third ingredient is a compound to be represented by a general formula similar to the above Formula VI. Because there is no $-(CH_2CH_2O)_nH$ chain in the third ingredient used in the formulation 1, it is an unstable composition. Accordingly, no whitening effect and no prevention of yellowing are recognized with respect to the formulation 1, and tinting phenomena are remarkable. The formulations 2, 3, 4, 6 and 8 relate to the ones in which the third ingredient belonging to the present invention is incorporated. The formulation 4 shows that the effect is smaller because the mole number of ethylene oxide is 14. The formulations 5 and 7 show that, when $n$ of the $-(CH_2CH_2O)_nH$ chain is more than 15, the substantivity of optical brighteners will be reduced, so that no whitening effect and no prevention of yellowing are obtained. The third ingredient having $-(CH_2CH_2O)_nH$ chain in which $n$ is more than 15 will show excellent effects in the stabilization of the solution and in reducing the tinting phenomenon. The formulations 6, 7 and 8 relate to examples having one $-(CH_2CH_2O)_nH$ chain, and the formulation 8 relates to an example having two long chain alkyl radicals.

(2) The formulations 9 to 13 are examples having a compound of the general Formula VII. The formulations 9 to 12 relate to examples having

$$-(CH_2CH_2O)_nH$$

chain in which $n$ is less than 15. The formulations 9 and 10 relate to examples having a kind of alkyl radical, respectively, the formulation 11 relates to an example having $R_{17}CONHCH_2CH_2-$, and the formulation 12 relates to an example having

$$R_{11}COOCH_2CH_2CH_2-$$

It is remarked that the formulation 13 relates to an example having an alkyl radical and, when

$$-(CH_2CH_2O)_nH$$

of this type of the third ingredient is unreasonably long, the substantivity of optical brightener is inferior.

(3) The formulations 14 and 15 relate to examples having a compound of the general Formula VIII. The formulation 14 relates to an example having an ethylene oxide chain of 10 moles and the formulation 15 relates to an example having an ethylene oxide chain of 20 moles. It should be noted that the ethylene oxide chain connected by $R_3$ is different from a chain having an hydroxy group on the end of the chain. This means that X and Y of the compound of the general Formula VIII must be $X+Y \leqq 14$.

(4) The formulations 16 to 18 relate to examples having a compound of the general Formula IX. The formulations 16 and 17 relate to examples having a third ingredient belonging to the present invention, respectively, and the formulation 18 relates to an example having no $-(CH_2CH_2O)_nH$ chain and it shows that the effects are inferior in such a case.

(5) The formulation 19 in Table 1 relates to an example having a compound of the general Formula X, and it shows that all of the effects are excellent.

(6) The formulations 20 and 21 relate to examples having compounds of the general Formula XI. The formulation 20 is excellent, but the formulation 21 shows that any remarkable effect is not obtained because of the presence of no $-(CH_2CH_2O)_nH$ chain.

(7) The formulation 22 in Table 1 shows that it does not have an effect of improving the substantivity of the optical brightener, because it contains a non-ionic surface active agent.

(8) The formulation 23 shows that, when only a cationic surface active agent of the general Formula I and an optical brightener of the general Formula V are blended, a precipitation occurs, so that there is no adsorption of the optical brightener on the fabrics.

(9) The formulations 24 to 26 shows that, even though a cationic surface active agent of the general Formula I is formulated with the third ingredient belonging to the present invention, there is no effect, if the optical brightener used is that of the general Formula II.

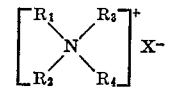
(10) The formulations 27 and 28 show that the formulation in the combination of a cationic active agent of the general Formula I, the third ingredient of the present invention and a cationic optical brightener of the general Formula III or IV, but it never adsorbs on cellulose textiles and there is no whitening effect.

(11) The formulation 29 shows that, when the optical brightener is that of the general Formula II and the third ingredient is a non-ionic surface active agent the composition has no action of stabilizing a complex of the optical brightener with a cationic surface active agent in the system, and the optical brightener does not adsorb on the fabrics. The formulation 30 shows that, when the optical brightener is that of the general Formula III and the third ingredient is a non-ionic surface active agent, the formulation is stable, but the optical brightener slightly adsorbs on fabrics.

What we claim is:

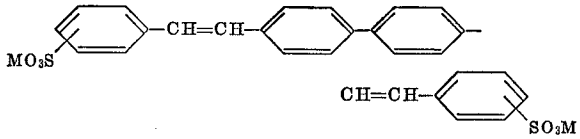
1. A textile treating composition consisting essentially of:

(1) 1 to 10 parts by weight of cationic surface active agent of the formula



wherein $R_1$ and $R_2$ are alkyls having 12 to 22 carbon atoms, $R_3$ and $R_4$ are alkyls having 1 to 4 carbon atoms, and X is halogen, $CH_3SO_4^-$ or $C_2H_5SO_4^-$

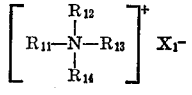
(2) 0.05 to 0.5 part by weight of optical brightener of the formula



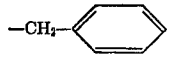
wherein M is alkali metal or ethanolamine, and

(3) 0.5 to 10 parts by weight of cationic surface active agent selected from the group consisting of

(a) a compound of the formula



wherein $R_{11}$ and $R_{12}$ are $-CH_3$, $-C_2H_5$,

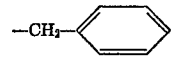


$-CH_2R_{15}$, $-(CH_2)_kNHCOR_{15}$ or $-(CH_2)_kOOCR_{15}$

(wherein $R_{15}$ is alkyl having 11 to 21 carbon atoms and $k$ is an integer of from 1 to 3) and either one of $R_{11}$ or $R_{12}$ is $-CH_2R_{15}$,

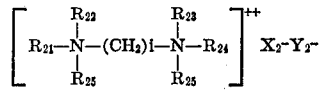
$-(CH_2)_kNHCOR_{15}$

or $-(CH_2)_kOOCR_{15}$; $R_{13}$ is $-CH_3$, $-C_2H_5$,



or $-(CH_2CH_2O)_mH$, $R_{14}$ is $-(CH_2CH_2O)_nH$, $m$ and $n$ are integers of more than one, and $2 \leqq m+n \leqq 14$, $X_1^-$ is halogen, $CH_3SO_4^-$ or $C_2H_5SO_4^-$;

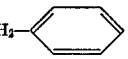
(b) a compound of the formula



wherein $R_{21}$ is $-CH_2R_{26}$, $-(CH_2)_kNHCOR_{26}$ or $-(CH_2)_kOOCR_{26}$, (wherein $R_{26}$ is alkyl having 11 to 21 carbon atoms and $k$ is an integer from 1 to ), $R_{22}$, $R_{23}$ and $R_{24}$ each is

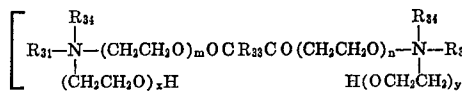
$-(CH_2CH_2O)_lH$,

$-(CH_2CH_2O)_mH$ or $-(CH_2CH_2O)_nH$, and $l$, $m$ and $n$ each is an integer of more than one, and $3 \leqq l+m+n \leqq 14$, $R_{25}$ is $-CH_3$, $-C_2H_5$ or

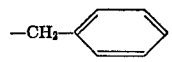


$X_2^-$ and $Y_2^-$ are halogen, $CH_3SO_4^-$ or $C_2H_5SO_4^-$ $i$ is an integer of from 2 to 3;

(c) a compound of the formula

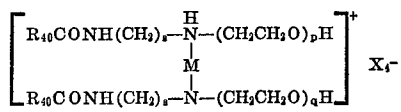


wherein $R_{31}$ and $R_{32}$ are alkyls having 12 to 21 carbon atoms, $R_{33}$ is alkylene having more than 2 carbon atoms, $R_{34}$ is $-CH_3$, $-C_2H_5$ or

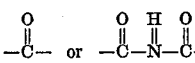


$m$ and $n$ are integers of more than one, and $2 \leqq m+n \leqq 14$, $x$ and $y$ are integers of more than one, and $2 \leqq x+y \leqq 14$, and $X_3^-$ and $Y_3^-$ are halogen, $CH_3SO_4^-$ or $C_2H_5CO_4^-$;

(d) a compound of the formula

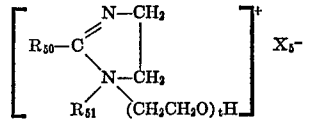


wherein M is

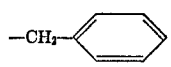


$R_{40}$ is alkyl having 12 to 21 carbon atoms, $s$ is 2 or 3, $p$ and $q$ are integers of more than one, and $2 \leqq p+q \leqq 14$, and $X_4^-$ is an anion obtained by removing one hydrogen from acetic acid, lactic acid, hydroxyacetic acid, boric acid or benzoic acid;

(e) a compound of the formula

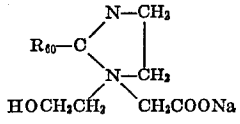


wherein $R_{50}$ is alkyl having 11 to 21 carbon atoms, $R_{51}$ is $-CH_3$, $-C_2H_5$ or



$t$ is an integer from 1 to 14, and $X_5^-$ is halogen, $CH_3SO_4^-$ or $C_2H_5SO_4^-$;

(f) a compound of the formula



wherein $R_{60}$ is alkyl having 11 to 21 carbon atoms.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HERBERT B. GUYNN, Primary Examiner

U.S. Cl. X.R.

117—33.5 T, 139.5 C, O; 252—8.7, 8.8, 301.2 W; 260—505 C

# UNITED STATES PATENT OFFICE
# CERTIFICATE OF CORRECTION

Patent No. 3 781 204 Dated December 25, 1973

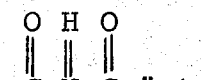
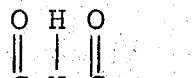
Inventor(s) Mamoru Katsumi, Toshio Sato and Tadao Hara

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

Col. 14, line 22; change "from 1 to )" to ---from 1 to 3)---.

Col. 14, line 32; change "$C_2H_5SO_4-$ i is" to ---$C_2H_5SO_4-$ , and i is---.

Col. 14, line 47; change "$C_2H_5CO_4-$" to ---$C_2H_5SO_4-$ ---.

Col. 14, line 56; change "  " to ---  ---.

**Signed and sealed this 18th day of June 1974.**

**(SEAL)**
**Attest:**

**EDWARD M.FLETCHER,JR.**
**Attesting Officer**

**C. MARSHALL DANN**
**Commissioner of Patents**

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