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(54) **LAUNDRY TREATMENT COMPOSITIONS**

WÄSCHEBEHANDLUNGSMITTEL

COMPOSITIONS DE TRAITEMENT DE LINGE

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(56) References cited:
WO-A-20/05003277 **US-A- 3 960 744**
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EP 1 794 274 B1

Description**TECHNICAL FIELD**

[0001] The present invention relates to laundry treatment compositions which comprise a dye.

BACKGROUND OF THE INVENTION

[0002] Reactive dyes are coloured compounds with one or more functional groups capable of forming a covalent bond with a suitable substrate, generally cotton or other cellulosic fibres. Typical reactive groups of the reactive dyes are monochlorotriazinyl, monofluorotriazinyl and 2-sulfooxyethylsulfonyl. Typical chromophores of the reactive dyes are azo, anthraquinone, phthalocyanine, formazan and triphenyldioxazine.

[0003] The reactive dyes have specific functional groups that can undergo addition or substitution reactions with -OH, -SH and -NH₂ groups present in textile fibers. A consequence of the nature of these specific functional groups is that reactive dyes can cause irritation/sensitization of the respiratory tract and skin. There is also evidence that they give rise to contact dermatitis, allergic conjunctivitis, rhinitis, occupational asthma and other allergic reactions.

[0004] US 3,960,744 discloses a powder detergent composition comprising a bluing dye precursor.

SUMMARY OF THE INVENTION

[0005] We have found that hydrolysed reactive dyes may be used to impart shading to textiles whilst reducing the risk of irritation/sensitization of the respiratory tract and skin in comparison to reactive dyes.

[0006] In the presence of water and high pH the reactive groups of reactive dyes are hydrolysed. We have found that the hydrolysed reactive dye is also substantive to cotton under normal wash conditions.

[0007] In one aspect the present invention provides a laundry treatment composition comprising between 0.0001 to 0.1 wt % of a hydrolysed reactive dye and between 2 to 60 wt % of a surfactant.

[0008] In another aspect the present invention provides a method of treating a textile, the method comprising the steps of: (i) treating a textile with an aqueous solution of a hydrolysed reactive dye, the aqueous solution comprising from 10 ppb to 1 ppm of the hydrolysed reactive dye and from 0.2 g/L to 3 g/L of a surfactant; and, (ii) rinsing and drying the textile. Most preferably the hydrolysed reactive dye is at a concentration in the range from 100 ppb to 500 ppb. The present invention also extends to the aqueous solution used in the method.

[0009] A "unit dose" as used herein is a particular amount of the laundry treatment composition used for a type of wash, conditioning or requisite treatment step. The unit dose may be in the form of a defined volume of powder, granules or tablet or unit dose detergent liquid.

DETAILED DESCRIPTION OF THE INVENTION

[0010] The reactive dyes may be considered to be made up of a chromophore which is linked to an anchoring moiety. The chromophore may be linked directly to the anchor or via a bridging group. The chromophore serves to provide a colour and the anchor to bind to a textile substrate.

[0011] A marked advantage of reactive dyes over direct dyes is that their chemical structure is much simpler, their absorption bands are narrower and the dyeing/shading are brighter; industrial Dyes, K. Hunger ed. Wiley-VCH 2003 ISBN 3-527-30426-6. However, mammalian contact with reactive dyes results in irritation and/or sensitisation of the respiratory tract and/or skin. In addition, wash conditions are not ideal for deposition of dyes because the efficiency of deposition is low.

[0012] With regard to reducing irritation and/or sensitisation, it is preferred that each individual anchor group of each reactive dyes is hydrolysed such that the most reactive group(s) of anchor groups of the dye is/are hydrolysed. In this regard, the term hydrolysed reactive dye encompasses both fully and partially hydrolysed reactive dyes.

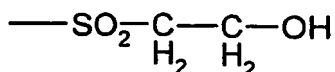
[0013] The reactive dye may have more than one anchor. If the dye has more than one anchor, then each and every anchor, that contributes to irritation and/or sensitisation, needs to be hydrolysed to the extent discussed above.

[0014] The dyes used in the present invention comprise a chromophore and an anchor that are covalently bound and may be represented in the following manner: Chromophore-anchor. The linking between the chromophore and an anchor are preferably provided by -NH-CO-, -NH-, NHCO-CH₂CH₂-, -NH-CO-, or -N=N-.

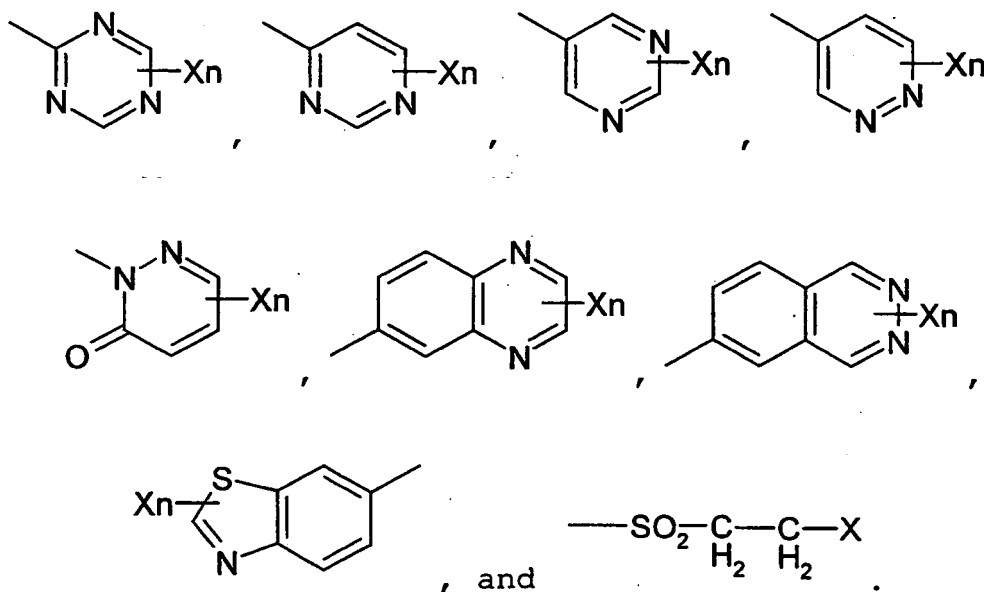
[0015] It is preferred that when a "unit dose" of the laundry treatment composition is dissolved in water the ionic strength of the resultant aqueous laundry treatment composition is between 0.001 to 0.5, more preferably between 0.02 to 0.2.

[0016] Preferably the hydrolysed reactive dye comprises a chromophore moiety covalently bound to an anchoring group, the anchoring group for binding to cotton, the anchoring group selected from the group consisting of: a heteroar-

omatic ring, preferably comprising a nitrogen heteroatom, having at least one -OH substituent covalently bound to the heteroaromatic ring, and



[0017] It is preferred that the anchor group is of the form:

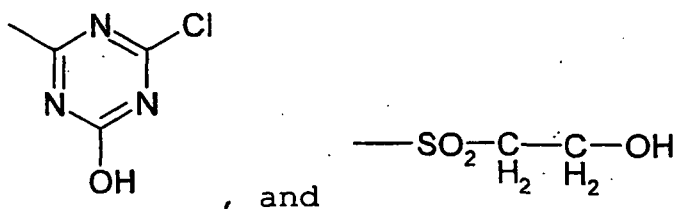


wherein:

n takes a value between 1 and 3:

X is selected from the group consisting of: -Cl, -F, NHR, a quaternary ammonium group, -OR and -OH;

R is selected from: an aromatic group, benzyl, a C1-C6-alkyl; and, wherein at least one X is -OH. It is preferred that R is selected from naphthyl, phenyl, and -CH₃. Most preferably the anchor group is selected from the group consisting of:



[0018] Preferably, the chromophore is selected from the group consisting of: azo, anthraquinone, phthalocyanine, formazan and triphenodioaxazine.

[0019] Preferably, the chromophore is linked to the hydrolysed anchor by a bridge selected from the group consisting of: -NH-CO-, -NH-, NHCO-CH₂CH₂-, -NH-CO-, and -N=N-.

[0020] Most preferred hydrolysed reactive dyes are hydrolysed Reactive Red 2, hydrolysed Reactive Blue 4, hydrolysed Reactive Black 5, and hydrolysed Reactive Blue 19.

BALANCE CARRIERS AND ADJUNCT INGREDIENTS

[0021] The laundry treatment composition, in addition to the hydrolysed reactive dye and surfactant, comprises the balance carriers and adjunct ingredients to 100 wt % of the composition.

[0022] These may be, for example, builders, foam agents, anti-foam agents, solvents, fluorescers, bleaching agents, and enzymes. The use and amounts of these components are such that the composition performs depending upon economics, environmental factors and use of the composition.

[0023] The composition comprises a surfactant and optionally other conventional detergent ingredients. The composition may also comprise an enzymatic detergent composition which comprises from 0.1 - 50 wt %, based on the total detergent composition, of one or more surfactants. This surfactant system may in turn comprise 0 - 95 wt % of one or more anionic surfactants and 5 to 100 wt % of one or more nonionic surfactants. The surfactant system may additionally contain amphoteric or zwitterionic detergent compounds, but this is not normally desired owing to their relatively high cost. The enzymatic detergent composition according to the invention will generally be used as a dilution in water of about 0.05 to 2 wt%.

[0024] The composition comprises between 2 to 60 wt % of a surfactant, preferably 10 to 30 wt %. In general, the nonionic and anionic surfactants of the surfactant system may be chosen from the surfactants described "Surface Active Agents" Vol. 1, by Schwartz & Perry, Interscience 1949, Vol. 2 by Schwartz, Perry & Berch, Interscience 1958, in the current edition of "McCutcheon's Emulsifiers and Detergents" published by Manufacturing Confectioners Company or in "Tenside-Taschenbuch", H. Stache, 2nd Edn., Carl Hauser Verlag, 1981.

[0025] Suitable nonionic detergent compounds which may be used include, in particular, the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Specific nonionic detergent compounds are C₆-C₂₂ alkyl phenol-ethylene oxide condensates, generally 5 to 25 EO, i.e. 5 to 25 units of ethylene oxide per molecule, and the condensation products of aliphatic C₈-C₁₈ primary or secondary linear or branched alcohols with ethylene oxide, generally 5 to 40 EO.

[0026] Suitable anionic detergent compounds which may be used are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals. Examples of suitable synthetic anionic detergent compounds are sodium and potassium alkyl sulphates, especially those obtained by sulphating higher C₈-C₁₈ alcohols, produced for example from tallow or coconut oil, sodium and potassium alkyl C₉-C₂₀ benzene sulphonates, particularly sodium linear secondary alkyl C₁₀-C₁₅ benzene sulphonates; and sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum. The preferred anionic detergent compounds are sodium C₁₁-C₁₅ alkyl benzene sulphonates and sodium C₁₂-C₁₈ alkyl sulphates. Also applicable are surfactants such as those described in EP-A-328 177 (Unilever), which show resistance to salting-out, the alkyl polyglycoside surfactants described in EP-A-070 074, and alkyl monoglycosides.

[0027] Preferred surfactant systems are mixtures of anionic with nonionic detergent active materials, in particular the groups and examples of anionic and nonionic surfactants pointed out in EP-A-346 995 (Unilever). Especially preferred is surfactant system that is a mixture of an alkali metal salt of a C₁₆-C₁₈ primary alcohol sulphate together with a C₁₂-C₁₅ primary alcohol 3-7 EO ethoxylate.

[0028] The nonionic detergent is preferably present in amounts greater than 10%, e.g. 25-90 wt % of the surfactant system. Anionic surfactants can be present for example in amounts in the range from about 5% to about 40 wt % of the surfactant system.

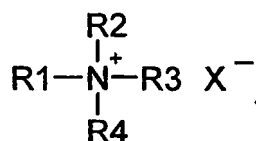
CATIONIC COMPOUND

[0029] When the present invention is used as a fabric conditioner it needs to contain a cationic compound.

[0030] Most preferred are quaternary ammonium compounds.

[0031] It is advantageous if the quaternary ammonium compound is a quaternary ammonium compound having at least one C₁₂-C₂₂ alkyl chain.

[0032] It is preferred if the quaternary ammonium compound has the following formula:



in which R¹ is a C₁₂ to C₂₂ alkyl or alkenyl chain; R², R³ and R⁴ are independently selected from C₁-C₄ alkyl chains and

X⁻ is a compatible anion. A preferred compound of this type is the quaternary ammonium compound cetyl trimethyl quaternary ammonium bromide.

[0033] A second class of materials for use with the present invention are the quaternary ammonium of the above structure in which R¹ and R² are independently selected from C₁₂ to C₂₂ alkyl or alkenyl chain; R³ and R⁴ are independently selected from C₁-C₄ alkyl chains and X⁻ is a compatible anion.

[0034] A detergent composition according to claim 1 in which the ratio of (ii) cationic material to (iv) anionic surfactant is at least 2:1.

[0035] Other suitable quaternary ammonium compounds are disclosed in EP 0 239 910 (Proctor and Gamble).

[0036] It is preferred if the ratio of cationic to nonionic surfactant is from 1:100 to 50:50, more preferably 1:50 to 20:50.

[0037] The cationic compound may be present from 0.02 wt % to 20 wt % of the total weight of the composition.

[0038] Preferably the cationic compound may be present from 0.05 wt % to 15 wt %, a more preferred composition range is from 0.2 wt % to 5 wt %, and most preferably the composition range is from 0.4 wt % to 2.5 wt % of the total weight of the composition.

[0039] If the product is a liquid it is preferred if the level of cationic surfactant is from 0.05wt % to 10wt % of the total weight of the composition. Preferably the cationic compound may be present from 0.2 wt % to 5 wt %, and most preferably from 0.4 wt % to 2.5 wt % of the total weight of the composition.

[0040] If the product is a solid it is preferred if the level of cationic surfactant is 0.05 wt % to 15 wt % of the total weight of the composition. A more preferred composition range is from 0.2 wt % to 10 wt %, and the most preferred composition range is from 0.9 wt % to 3.0 wt % of the total weight of the composition.

BLEACHING SPECIES

[0041] The laundry treatment composition may comprise bleaching species. The bleaching species, for example, may be selected from perborate and percarbonate. These peroxy species may be further enhanced by the use of an activator, for example, TAED or SNOBS. Alternatively or in addition to, a transition metal catalyst may be used with the peroxy species. A transition metal catalyst may also be used in the absence of peroxy species where the bleaching is termed to be via atmospheric oxygen, see, for example WO02/48301. Photobleaches, including singlet oxygen photobleaches, may be used with the laundry treatment composition. A preferred photobleach is vitamin K.

FLUORESCENT AGENT

[0042] The laundry treatment composition preferably comprises a fluorescent agent(optical brightener). Fluorescent agents are well known and many such fluorescent agents are available commercially. Usually, these fluorescent agents are supplied and used in the form of their alkali metal salts, for example, the sodium salts. The total amount of the fluorescent agent or agents used in laundry treatment composition is generally from 0.005-2 wt %, more preferably 0.01 to 0.1 wt %. Preferred classes of fluorescer are: Di-styryl biphenyl compounds, e.g. Tinopal (Trade Mark) CBS-X, Diamine stilbene di-sulphonic acid compounds, e.g. Tinopal DMS pure Xtra and Blankophor (Trade Mark) HRH, and Pyrazoline compounds, e.g. Blankophor SN. Preferred fluorescers are: sodium 2 (4-styryl-3-sulfophenyl)-2H-naphthol [1,2-d]triazole, disodium 4,4'-bis[[(4-anilino-6-(N methyl-N-2 hydroxyethyl) amino 1,3,5-triazin-2-yl)]amino]stilbene-2-2' disulfonate, disodium 4,4'-bis[[(4-anilino-6-morpholino-1,3,5-triazin-2-yl)]amino] stilbene-2-2' disulfonate, and disodium 4,4'-bis(2-sulfostyryl)biphenyl.

EXAMPLES

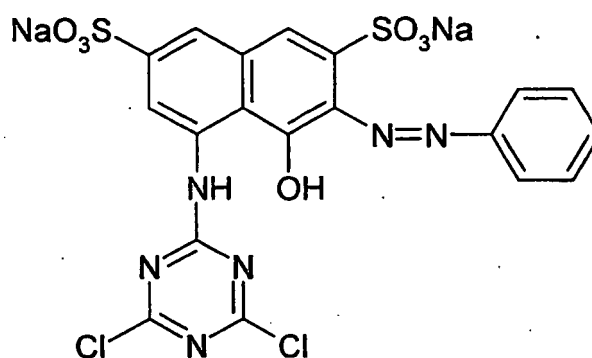
Example 1: Deposition on cotton

[0043] To determine the substantivity of a dye the following experiment was performed. A 0.1 wt % solution of the reactive dye was created in a pH = 10 buffer and was left for four days in the dark at room temperature to allow the reactive groups to hydrolyse. A stock solution of 1.5 g/L of a base washing powder in water was created. The washing powder contained 18 % NaLAS, 73 % salts (silicate, sodium tri-poly-phosphate, sulphate, carbonate), 3 % minors including perborate, fluorescer and enzymes, remainder impurities and water. The solution was divided into 60 ml aliquots and hydrolysed reactive dye added to this to give a solution of optical density of approximately one (5 cm pathlength) at the maximum absorption of the dye in the visible lengths, 400 to 700nm. The optical density was measured using a UV-visible spectrometer. One piece of bleached, non-mercerised, non-fluorescent woven cotton cloth (ex Phoenic Calico) weighing 1.3 g was placed in the solution at room temperature (20 °C). This cloth represents a slightly yellow cotton. The cloth was left to soak for 45 minutes then the solution agitated for 10 min., rinsed and dried. Following this the optical density of the solution was re-measured and the amount of dye absorbed by the cloth calculated.

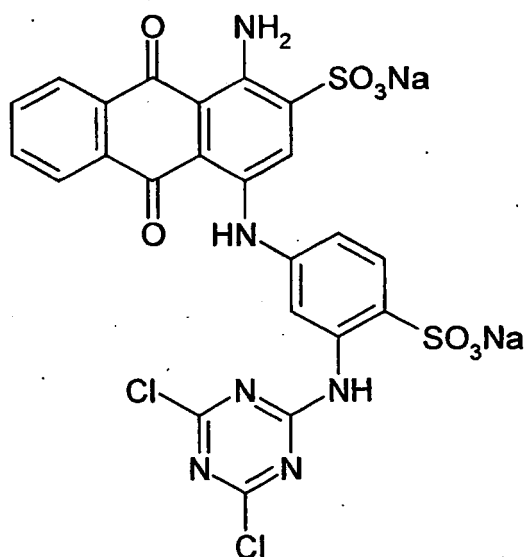
[0044] The results are given in the table below.

Dye	% deposited on cotton
Hydrolysed Reactive Red 2	11.6
Hydrolysed Reactive Blue 4	17.9
Hydrolysed Reactive Black 5	18.3
Hydrolysed Reactive Blue 19	22.0

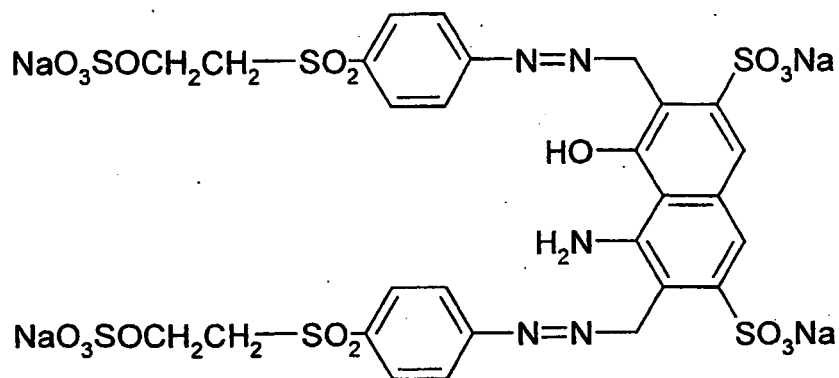
[0045] Reactive Red 2 [17804-49-8] represents a dichlorotriazinyl azo dye having the following structure:



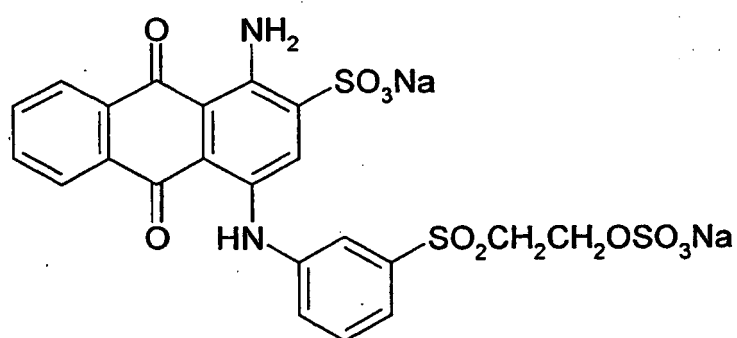
[0046] Reactive Blue 4 [13324-20-4] represents a dichlorotriazinyl anthraquinone dye having the following structure:



[0047] Reactive Black 5 [17095-24-8] represents a two anchor vinyl sulfonyl azo dye having the following structure:



[0048] Reactive Blue 19 [2580-78-1] represents a vinyl sulfonyl anthraquinone dye having the following structure:



[0049] All dyes were found to be substantive to cotton.

Example 2: Deposition on nylon

[0050] Deposition onto nylon fabric was measured in an analogous manner to Example 1, except nylon was used a fabric.

[0051] The results are given in the table below.

Dye	% deposited on nylon
Hydrolysed Reactive Red 2	0.7
Hydrolysed Reactive Blue 4	2.3
Hydrolysed Reactive Black 5	4.0
Hydrolysed Reactive Blue 19	5.60

[0052] Some of the hydrolysed dyes were found to be substantive to nylon.

Claims

1. A laundry treatment composition comprising between 0.0001 to 0.1 wt % of a hydrolysed reactive dye and between 2 to 60 wt % of a surfactant.
2. A laundry treatment composition according to claim 1, wherein the hydrolysed reactive dye comprises a chromophore moiety covalently bound to an anchoring group, the anchoring group for binding to cotton, the anchoring group

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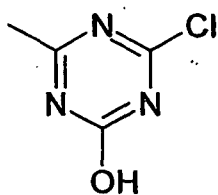


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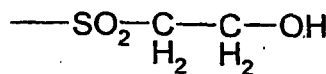
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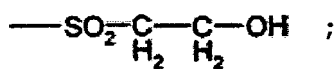
, and



8. A laundry treatment composition according to any one of claims 2 to 5, wherein the chromophore is linked to the hydrolysed anchor by a bridge selected from the group consisting of: -NH-CO-, -NH-, NHCO-CH₂CH₂-, -NH-CO-, and -N=N-.
9. A laundry treatment composition according to claim 1, wherein the hydrolysed reactive dye is selected from the hydrolysed product of: reactive red 2, reactive blue 4, reactive black 5, and reactive blue 19.
10. A laundry treatment composition according to any preceding claim, wherein the laundry treatment composition comprises from 0.005 to 2 wt % of a fluorescer.
11. A method of treating a textile, the method comprising the steps of:
- (i) treating a textile with an aqueous solution of a hydrolysed reactive dye, the aqueous solution comprising from 10 ppb to 1 ppm of the hydrolysed reactive dye and from 0.2 g/L to 3 g/L of a surfactant; and
 - (ii) rinsing and drying the textile.
12. A method of treating a textile according to claim 11, wherein the aqueous solution has an ionic strength from 0.001 to 0.5.

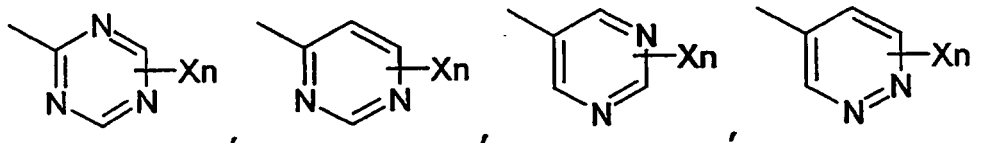
Patentansprüche

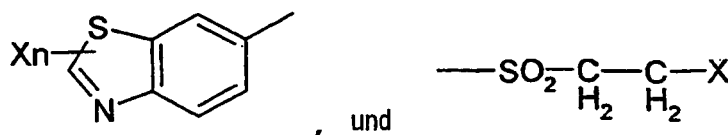
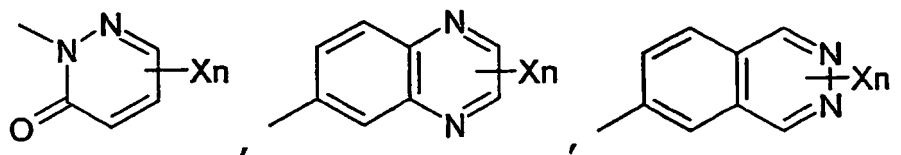
1. Wäschebehandlungszusammensetzung, umfassend zwischen 0,0001 bis 0,1 Gew.-% von einem hydrolysierten Reaktivfarbstoff und zwischen 2 bis 60 Gew.-% von einem Tensid.
2. Wäschebehandlungszusammensetzung nach Anspruch 1, worin der hydrolysierte Reaktivfarbstoff eine kovalent an eine Ankergruppe gebundene Chromophoreneinheit, die Ankergruppe zum Binden an Baumwolle, die Ankergruppe ausgewählt aus der Gruppe, bestehend aus: einem heteroaromatischen Ring mit mindestens einem Substituenten -OH, der kovalent an den heteroaromatischen Ring gebunden ist, und



umfasst.

3. Wäscheformulierung nach Anspruch 2, worin der heteroaromatische Ring ein Stickstoffheteroatom umfasst.
4. Wäscheformulierung nach Anspruch 3, worin die Ankergruppe von der Form:





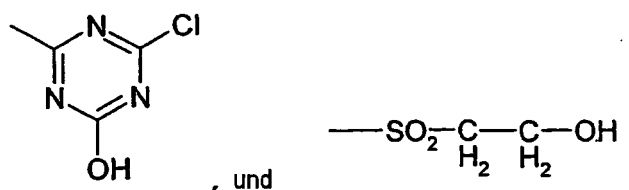
worin

n einen Wert zwischen 1 und 3 annimmt;

X ausgewählt ist aus der Gruppe bestehend aus: -Cl, -F, NHR, einer quaternären Ammoniumgruppe, -OR und -OH;

R ausgewählt ist aus: einer aromatischen Gruppe, Benzyl, einem C₁-C₆-Alkyl; und worin mindestens ein X -OH darstellt.

5. Wäschebehandlungszusammensetzung nach Anspruch 4, worin R aus Naphthyl, Phenyl und -CH₃ ausgewählt ist.
6. Wäschebehandlungszusammensetzung nach einem der Ansprüche 2 bis 5, worin das Chromophor aus der Gruppe, bestehend aus: Azo, Anthrachinon, Phthalocyanin, Formazan und Triphenodioaxazin, ausgewählt ist.
7. Wäschebehandlungszusammensetzung nach einem der Ansprüche 2 bis 5, worin die Ankereinheit ausgewählt ist aus der Gruppe bestehend aus:



8. Wäschebehandlungszusammensetzung nach einem der Ansprüche 2 bis 5, worin das Chromophor an den hydrolysierten Anker durch eine Brücke, ausgewählt aus der Gruppe, bestehend aus: NH-CO-, NH-, NHCO-CH₂CH₂-, NH-CO- und -N=N-, gebunden ist.
9. Wäschebehandlungszusammensetzung nach Anspruch 1, worin der hydrolysierte Reaktivfarbstoff ausgewählt ist aus dem hydrolysierten Produkt von: Reactive Red 2, Reactive Blue 4, Reactive Black 5 und Reactive Blue 19.
10. Wäschebehandlungszusammensetzung nach einem vorangehenden Anspruch, worin die Wäschebehandlungszusammensetzung 0,005 bis 2 Gew.-% von einem Fluoreszenzaufhellungsmittel umfasst.
11. Verfahren zum Behandeln eines Textils, wobei das Verfahren die Schritte umfasst von:

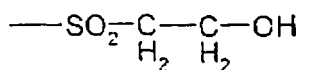
(i) Behandeln eines Textils mit einer wässrigen Lösung von einem hydrolysierten Reaktivfarbstoff, wobei die wässrige Lösung 10 ppb bis 1 ppm des hydrolysierten Reaktivfarbstoffs und 0,2 g/l bis 3 g/l von einem Tensid umfasst, und

(ii) Spülen und Trocknen des Textils.

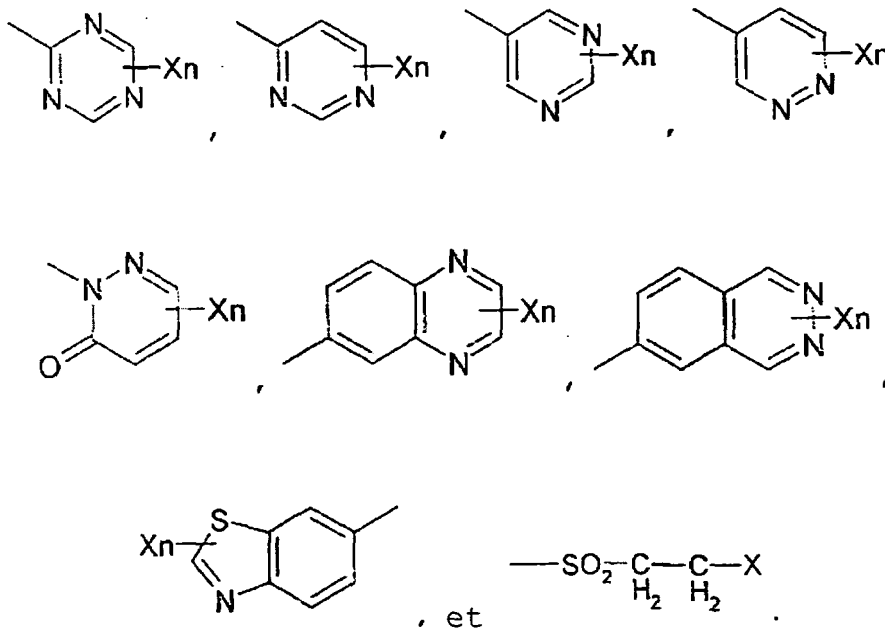
12. Verfahren zum Behandeln eines Textils nach Anspruch 11, wobei die wässrige Lösung eine Ionenstärke von 0,001 bis 0,5 aufweist.

Revendications

- Composition destinée au traitement du linge comprenant de 0,0001 à 0,1 % en poids d'un colorant réactif hydrolysé et de 2 à 60 % en poids d'un agent de surface.
- Composition destinée au traitement du linge selon la revendication 1, dans laquelle le colorant réactif hydrolysé comprend une entité de chromophore liée de manière covalente à un groupe d'ancrage, le groupe d'ancrage étant destiné à une liaison sur le coton, le groupe d'ancrage étant choisi dans le groupe comprenant : un noyau hétéroaromatique ayant au moins un substituant -OH lié de manière covalente au noyau hétéroaromatique, et



- Formulation de lessive selon la revendication 2, dans laquelle le noyau hétéroaromatique comprend un hétéroatome d'azote.
- Formulation de lessive selon la revendication 3, dans laquelle le groupe d'ancrage est de la forme :



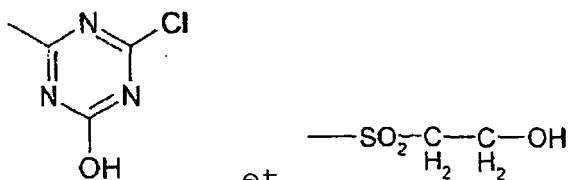
où :

n prend une valeur entre 1 et 3 ;
 X est choisi dans le groupe comprenant : -Cl, -F, NHR, un groupe ammonium quaternaire, -OR et -OH ;
 R est choisi parmi : un groupe aromatique, un benzyle, un alkyle en $C_1\text{--}C_6$; et, dans laquelle au moins un X est -OH.

- Composition destinée au traitement du linge selon la revendication 4, dans laquelle R est choisi parmi le naphthyle, le phényle, et -CH₃.

6. Composition destinée au traitement du linge selon l'une quelconque des revendications 2 à 5, dans laquelle le chromophore est choisi dans le groupe comprenant : l'azo, l'anthraquinone, la phtalocyanine, le formazan et la triphendioxazine.

7. Composition destinée au traitement du linge selon l'une quelconque des revendications 2 à 5, dans laquelle l'entité d'ancrage est choisie dans le groupe comprenant :



8. Composition destinée au traitement du linge selon l'une quelconque des revendications 2 à 5, dans laquelle le chromophore est lié à l'ancre hydrolysée par un pont choisi dans le groupe comprenant : -NH-CO-, -NH-, NHCO-CH₂CH₂-, -NH-CO-, et -N=N-.

9. Composition destinée au traitement du linge selon la revendication 1, dans laquelle le colorant réactif hydrolysé est choisi parmi le produit hydrolysé de : rouge réactif 2, bleu réactif 4, noir réactif 5, et bleu réactif 19.

10. Composition destinée au traitement du linge selon l'une quelconque des revendications précédentes, dans laquelle la composition de traitement du linge comprend de 0,005 à 2 % en poids d'un agent fluorescent.

11. Procédé de traitement d'un textile, le procédé comprenant les étapes consistant à :

- (i) traiter un textile avec une solution aqueuse d'un colorant réactif hydrolysé, la solution aqueuse comprenant de 10 ppb à 1 ppm du colorant réactif hydrolysé et de 0,2 g/l à 3 g/l d'un agent de surface ; et,
- (ii) rincer et sécher le textile.

12. Procédé de traitement d'un textile selon la revendication 11, dans laquelle la solution aqueuse a une force ionique allant de 0,001 à 0,5.

REFERENCES CITED IN THE DESCRIPTION

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