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(54) **A LIQUID HARD SURFACE ANTIMICROBIAL CLEANING COMPOSITION**

FLÜSSIGE ANTIMIKROBIELLE REINIGUNGSZUSAMMENSETZUNG FÜR HARTE OBERFLÄCHEN  
COMPOSITION LIQUIDE DE NETTOYAGE ANTIMICROBIEN DE SURFACES DURES

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**Description****Technical field**

5     **[0001]** The invention relates to a liquid hard surface cleaning and disinfection composition. The invention more particularly relates to a hard surface disinfection composition that provides synergistic antibacterial efficacy.

**Background of the invention**

10    **[0002]** Liquid compositions for disinfecting and cleaning hard surfaces like floors, walls, windows, doors, furniture, and table tops in various indoor and outdoor locations are known. Toilets and bathrooms are other places where germs proliferate and disinfection and cleaning compositions in liquid form are widely used. Other types of hard surfaces that are cleaned by such compositions include utensils and other articles found in households, offices and other public places.

15    **[0003]** Two of the popular classes of disinfecting / sanitizing actives are (i) bleaches and (ii) phenolics. Halogen and active oxygen are two most effective bleaches. Bleaches are compounds that liberate oxidising agents like chlorine, bromine, fluorine, nascent oxygen or other oxidizing agents that break the cell walls of microbes like bacteria and virus thereby disinfecting the surfaces thus treated. Phenolics are another class of antimicrobials often derived from essential oils extracted from natural sources which are formulated in an emulsified form and sold as floor and toilet cleaners.

20    **[0004]** These compounds are sometimes considered either harsh or take a long time for the antimicrobial action to take place. Very often, these compositions include surfactants to either emulsify the oily components present therein or to enable cleaning via detergency action or both. To remove the residual surfactants the surfaces being treated or cleaned are soon thereafter rinsed or wiped to be substantially free of the disinfection/ cleaning composition. This allows very little time for the disinfection action to take place.

25    **[0005]** Therefore, there is a need of providing a composition that gives relatively more efficacious antimicrobial action when cleaning period is relatively small, typically about 5 minutes or less, preferably lesser than 2 minutes and in many cases less than one minute or sometimes as low as 15 seconds or lesser. The present applicants have, in a prior patent application published as WO2010046238 disclosed a novel combination of thymol and terpineol that interact synergistically to enable fast antimicrobial action in less than about 15 seconds. The present inventors have found that inclusion of benzalkonium chloride and selected salts of di or tricarboxylic acid when combined with thymol or terpineol provides similar synergistic antimicrobial action in a rapid fashion and such efficacy is obtained at much lower concentrations of thymol and/ or terpineol. This invention has the advantage that essential oil actives like terpineol and thymol have a strong medicinal odour which is not liked by some consumers and it is advantageous to include thymol and terpineol at lower concentrations while ensuring that same efficient anti-microbial action. Further, it has been found by way of the present invention that inclusion of a chelating agent sodium tripolyphosphate provides for further enhanced antibacterial efficacy.

35    **[0006]** Liquid hard surface cleaning compositions having a pH 9 to 12 comprising cationic surfactant are known.

**[0007]** US6048836 (1998, P&G) discloses the use, in a composition, of a combination of a chelating agent, a surfactant, and an essential oil or an active thereof, to provide disinfecting properties.

40    **[0008]** The patent publication cited above does not address the problem of slow-acting antimicrobial composition, nor disclose the synergistic combination of actives disclosed and claimed in the present invention for the fast acting antimicrobial action in disinfecting hard surfaces. US 2009/0105195 also discloses antimicrobial compositions.

**[0009]** It is an object of the present invention to overcome or ameliorate at least one of the disadvantages of the prior art, or to provide a useful alternative.

45    **[0010]** Another object of the present invention is to provide a liquid hard surface cleaning and disinfecting composition that has relatively fast antimicrobial action.

**Summary of the invention**

50    **[0011]** According to the first aspect of the invention there is provided a liquid hard surface antimicrobial cleaning composition with a pH in the range of 9 to 12 comprising

- (a) 0.5 to 4% by weight of benzalkonium chloride;
- (b) 0.25 to 3% by weight of salt of di or tri carboxylic acid;
- (c) 0.01 to 2% by weight an essential oil antimicrobial active selected from thymol or terpineol; and
- 55    (d) 87 to 97% by weight water.

**[0012]** According to another aspect of the invention there is provided a method of cleaning and disinfecting a hard surface comprising applying to said hard surface a composition of the first aspect diluted with water in a weight ratio of

1: 20 to 1: 200.

**Detailed description of the invention**

[0013] These and other aspects, features and advantages will become apparent to those of ordinary skill in the art from a reading of the following detailed description and the appended claims. For the avoidance of doubt, any feature of one aspect of the present invention may be utilised in any other aspect of the invention. The word "comprising" is intended to mean "including" but not necessarily "consisting of" or "composed of." In other words, the listed steps or options need not be exhaustive. It is noted that the examples given in the description below are intended to clarify the invention and are not intended to limit the invention to those examples per se. Similarly, all percentages are weight/weight percentages unless otherwise indicated.

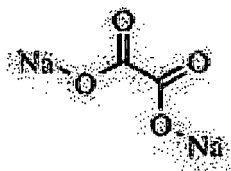
[0014] Except in the operating and comparative examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts of material or conditions of reaction, physical properties of materials and/or use are to be understood as modified by the word "about". Unless specified otherwise, numerical ranges expressed in the format "from x to y" are understood to include x and y. When for a specific feature multiple preferred ranges are described in the format "from x to y", it is understood that all ranges combining the different endpoints are also contemplated.

[0015] The first aspect of the invention provides for a liquid hard surface antimicrobial cleaning composition with a pH in the range of 9 to 12. The composition comprises 0.5 to 4% by weight of benzalkonium chloride; 0.25 to 3% by weight of salt of di or tri carboxylic acid; 0.01 to 2% by weight of an essential oil active selected from thymol or terpineol; and 87 to 97% by weight water.

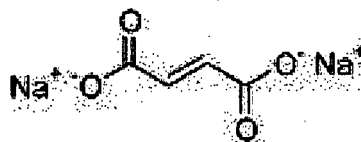
[0016] The composition comprises 0.5 to 4%, more preferably 1 to 2%, further more preferably 1.25 to 1.75% cationic surfactant benzalkonium chloride.

[0017] The composition comprises 0.25 to 3%, preferably 0.5 to 2%, more preferably 0.5 to 1.5% by weight of the composition salt of di or tri carboxylic acid. The salt of di or tri carboxylic acid is preferably chosen from an oxalate, fumarate, phthalate, maleate, malate, malonate or citrate. The salt of di or tricarboxylic acid is most preferably a phthalate, malate, malonate, or citrate. Alkali metal or alkaline earth metal salts are preferred, more preferably alkali metal salts of which sodium salt is most preferred. Structure of salts of some of the carboxylic acids are given below:

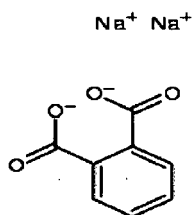
Sodium oxalate



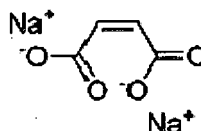
Sodium fumarate



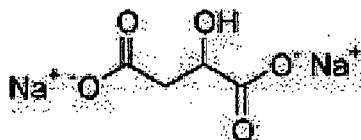
Sodium phthalate



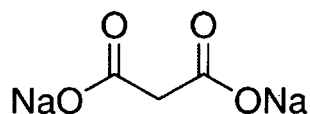
Sodium maleate



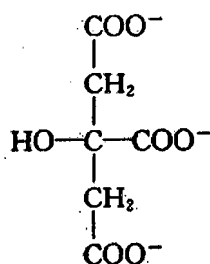
Sodium malate



Sodium malonate



Citrate

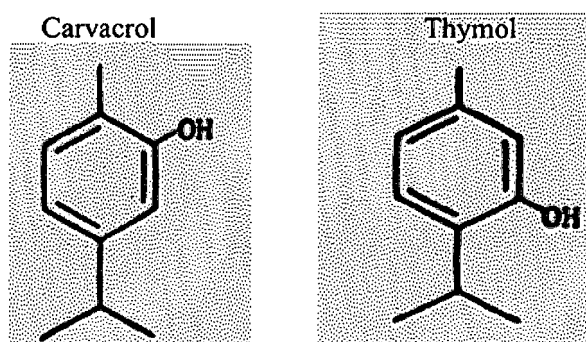


**[0018]** It is observed that inclusion of di or tri carboxylates in the composition of the invention provides for the desired antimicrobial efficacy while mono carboxylates do not demonstrate as good an efficacy.

**[0019]** The composition comprises an essential oil antimicrobial active selected from thymol or terpineol at 0.01 to 2% by weight of the composition.

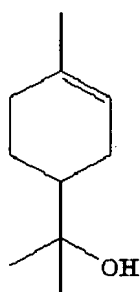
**[0020]** Preferred antimicrobial compositions of the present invention have thymol higher than 0.05 and lesser than 0.3% by weight. Thymol may be added to the antimicrobial composition in purified form. Alternatively, thyme oil or thyme extract comprising thymol may be added to the antimicrobial composition, while ensuring that thymol is present in the desired concentration in the composition of the present invention. Thyme oil or thyme extract is obtained from the thyme plant. Thyme plant refers to a plant belonging to genus *Thymus* and includes but is not limited to the following species: *Thymus vulgaris*, *Thymus zygis*, *Thymus saturoides*, *Thymus mastichina*, *Thymus broussonetti*, *Thymus maroccanus*, *Thymus pallidus*, *Thymus algeriensis*, *Thymus serpyllum*, *Thymus pulegoide*, and *Thymus citriodorus*.

**[0021]** The structures of thymol and its isomer carvacrol are given below:



**[0022]** Preferred antimicrobial compositions of the present invention have terpineol higher than 0.05 and lesser than 1% by weight. The terpineol is preferably selected from alpha-terpineol, beta-terpineol, gamma-terpineol or mixtures thereof. It is particularly preferred that the terpineol is alpha-terpineol. Terpineol may be added to the antimicrobial composition in purified form. Alternatively pine oil comprising terpineol may be added to the antimicrobial composition.

**[0023]** The structure of a terpineol compound is given below:



**[0024]** The composition optionally comprises sodium tripoly phosphate (STPP). When present, STPP is present in 0.25 to 3%, preferably 0.5 to 2%, more preferably 1.25 to 1.75% by weight of the composition.

**[0025]** The composition of the invention comprises water as a preferred carrier. Water is present in 87 to 97%, preferably

90 to 95% by weight of the composition.

**[0026]** A composition of the invention optionally comprises a surfactant. Preferred surfactant is a non-ionic surfactant. Non-ionic surfactant is preferably present in an amount of 2 to 10% by weight of the composition. Particularly preferred nonionic surfactants are fatty alcohol ethoxylates preferably C8-C22, more preferably C8-C16 fatty alcohol ethoxylates, comprising between 1 and 8 ethylene oxide groups. Suitable surfactant concentrations in liquid form of hard surface cleaning compositions are preferably from 1 to 5 % by weight of the composition.

**[0027]** The composition of the invention has pH in the range of 9 to 12, preferably in the range of 10.5 to 11.5. This could be achieved by incorporating the essential ingredients of the invention in selected amounts, else may be achieved by incorporating an alkaline agent. The alkaline agent is preferably sodium carbonate or sodium hydroxide. The alkaline agent is preferably present in 0.1 to 1% by weight of the composition.

**[0028]** The present inventors have determined that the highly efficacious antimicrobial action of the composition of the invention can be achieved in the absence of conventional disinfectants like bleaches e.g. halogens, halogenated compounds and active oxygen based bleaches. However small amount of such bleaches may be present in the composition e.g. less than 1%, preferably less than 0.5%, more preferably less than 0.1% and the efficacy of the composition is achievable even with the bleaches being absent from the composition.

**[0029]** The composition may further comprise various additional ingredients known to a person skilled in the art. Such additional ingredients include but are not limited to: perfumes, pigments, preservatives, emollients, sunscreens, emulsifiers, gelling agents, or thickening agents.

**[0030]** The antimicrobial composition may be in form of a liquid but the liquid composition may be further thickened to a gel or a paste. The antimicrobial composition of the present invention can be used for cleansing and care of hard surfaces such as glass, metal, plastic, wood, ceramic and the like.

**[0031]** According to another aspect of the present invention there is provided a method of cleaning and disinfecting a hard surface comprising applying to said hard surface a composition of the invention diluted with water in a weight ratio of 1: 20 to 1: 200, preferably in a ratio of 1:40 to 1:80.

**[0032]** The method preferably comprises a step of rinsing the surface with a suitable solvent preferably water or the surface may be wiped with a suitable wipe.

**[0033]** The inventors have determined that the composition of the invention provides an antimicrobial action where the contact time of the antimicrobial actives with the surface is low, i.e. of the order of less than 5 minutes, preferably less than 2 minutes, further more preferably less than a minute and in many cases less than 15 seconds.

**[0034]** The invention will now be illustrated with the help of the following non-limiting examples.

## Examples

### Examples 1 to 9: Antibacterial efficacy of the composition of the invention

**[0035]** Antibacterial compositions for hard surface cleaning as shown in Table - 1 read along with Table - 2 were prepared. The compositions were tested for antibacterial efficacy using the European Suspension Test BS EN1276 which is briefly described below:

A test suspension of bacteria (1 ml) was added to a sample of the product under a certain dilution condition (8 ml) along with a specific soil (1 ml). The mixture was maintained at 20°C for 5 minutes. After this period of contact time, an aliquot was taken and was immediately neutralized by a validated method. The number of bacteria in each sample were determined by plating in TSA agar and the reduction in viable count was calculated after incubating for 48 hours.

**[0036]** In Table - 1, below the organism used was bacteria *P. aeruginosa* ATCC 15442 (at 10<sup>7</sup> cfu/ml concentration); the dilution was 1:60 of the product (a floor cleaner) in hard water. The soil used was used was Bovine Serum Albumin (BSA) at a final concentration of 0.03%. The pH of the samples was 11.0.

Table - 1

| Ingredients                              | Weight%               |
|------------------------------------------|-----------------------|
| Neodol (C12E09)                          | 3.0                   |
| Benzalkonium chloride                    | 1.5                   |
| Essential oil active                     | As shown in Table - 2 |
| Sodium Carbonate                         | 0.3                   |
| Di/tri carboxylate (as shown in Table 2) | 1.0                   |

Table - 2: Compositions as per Table - 1 where prepared using the di/carboxylic acids and thymol and/or terpineol concentrations as shown below:

| Example    | Di/tri carboxylic acid | Thymol (wt%) | Terpineol (wt%) | Log reduction |
|------------|------------------------|--------------|-----------------|---------------|
| Example 1  | -                      | -            | -               | 3.2           |
| Example 2  | -                      | 0.125        | -               | 3.1           |
| Example 3  | Sodium malonate        | 0.125        | -               | 6.4           |
| Example 4  | Sodium citrate         | 0.125        | -               | 7.3           |
| Example 5  | -                      | -            | 0.25            | 3.0           |
| Example 6  | Sodium malonate        | -            | 0.25            | 5.2           |
| Example 7  | Sodium citrate         | -            | 0.25            | 7.3           |
| Example 8  | -                      | 0.125        | 0.25            | 3.3           |
| Example 9  | Sodium malonate        | 0.125        | 0.25            | 6.8           |
| Example 10 | Sodium citrate         | 0.125        | 0.25            | 7.3           |

**[0037]** The data in Table 1 and 2 indicates that the compositions as per the invention (Examples 3,4,6,7,9,10) provide for vastly superior antibacterial efficacy as compared to compositions outside the invention (Examples 1,2,5,8).

Examples 11 to 13:

**[0038]** Experiments similar to Examples 1 to 10 were carried out except that the dilution was 1:40 and the time of contact of the product with the bacteria was 30 seconds instead of 5 minutes. The data is summarized in Table - 3.

Table - 3:

| Ingredients               | Example 11 (wt%) | Example 12 (wt%)    | Example 13 (wt%)   |
|---------------------------|------------------|---------------------|--------------------|
| Neodol (C12E09)           | 3.0              | 3.0                 | 3.0                |
| Benzalkonium chloride     | 1.5              | 1.5                 | 1.5                |
| Sodium Carbonate          | 0.3              | 0.3                 | 0.3                |
| Di/tri carboxylate        | -                | Sodium malonate 1.0 | Sodium citrate 1.0 |
| Thymol                    | 0.125            | 0.125               | 0.125              |
| Terpineol                 | 0.25             | 0.25                | 0.25               |
| Log reduction of bacteria | 2.0              | 5.2                 | 6.8                |

**[0039]** The data in Table 3 indicates that the antibacterial compositions of the invention (Examples 12 and 13) are effective even at short contact times of about 30 seconds.

Examples 14 to 19: Antibacterial efficacy of compositions of the invention with various di/tricarboxylates

**[0040]** Compositions as shown in Table - 4 were tested for antibacterial efficacy at a dilution of 1:60 with hard water at a contact time of 5 minutes. The soil was BSA at 0.3%. The bacteria used was *P. aeruginosa* ATCC 15442 (at  $10^7$  cfu/ml cell density). The pH of the compositions were 11. The data is shown in Table - 4.

Table -4

| Ingredients                       | Example 14<br>(wt%) | Example 15<br>(wt%)    | Example 16<br>(wt%)     | Example 17<br>(wt%)  | Example 18<br>(wt%)    | Example 19<br>(wt%)   |
|-----------------------------------|---------------------|------------------------|-------------------------|----------------------|------------------------|-----------------------|
| Neodol<br>(C12EO9)                | 3.0                 | 3.0                    | 3.0                     | 3.0                  | 3.0                    | 3.0                   |
| Benzalkonium<br>chloride          | 1.5                 | 1.5                    | 1.5                     | 1.5                  | 1.5                    | 1.5                   |
| STPP*                             | 1.5                 | 1.5                    | 1.5                     | 1.5                  | 1.5                    | 1.5                   |
| Sodium<br>Carbonate               | 0.3                 | 0.3                    | 0.3                     | 0.3                  | 0.3                    | 0.3                   |
| Di / tri<br>carboxylate           | -                   | Sodium<br>fumarate 1.0 | Sodium<br>phthalate 1.0 | Sodium<br>malate 1.0 | Sodium<br>malonate 1.0 | Sodium<br>citrate 1.0 |
| Thymol                            | 0.125               | 0.125                  | 0.125                   | 0.125                | 0.125                  | 0.125                 |
| Terpineol                         | 0.25                | 0.25                   | 0.25                    | 0.25                 | 0.25                   | 0.25                  |
| Log reduction<br>of bacteria      | 4.3                 | 5.2                    | 7.3                     | 5.9                  | 7.3                    | 7.3                   |
| *STPP: is sodium tripolyphosphate |                     |                        |                         |                      |                        |                       |

**[0041]** The data in Table 4 indicates that various other di/tri carboxylates may be used in the composition of the invention in the presence of essential oil actives and these samples (Examples 15 to 19) give vastly superior antibacterial efficacy as compared to a sample outside the invention (Example 14).

## Claims

1. A liquid hard surface antimicrobial cleaning composition with a pH in the range of 9 to 12 comprising
  - (a) 0.5 to 4% by weight of benzalkonium chloride;
  - (b) 0.25 to 3% by weight of salt of di or tri carboxylic acid;
  - (c) 0.01 to 2% by weight an essential oil antimicrobial active selected from thymol or terpineol; and
  - (d) 87 to 97% by weight water.
2. A composition as claimed in claim 1 comprising thymol and terpineol.
3. A composition as claimed in claim 1 or 2 wherein said salt of di or tri carboxylic acid is chosen from an oxalate, fumarate, phthalate, maleate, malate, malonate or citrate.
4. A composition as claimed in claim 3 wherein said salt of di or tricarboxylic acid is chosen from a phthalate, malate, malonate, or citrate.
5. A composition as claimed in any one of the preceding claims comprising 0.25 to 3% sodium tripolyphosphate.
6. A composition as claimed in any one of the preceding claims comprising a non-ionic surfactant.
7. A composition as claimed in claim 6 wherein said non-ionic surfactant is present in an amount of 2 to 10% by weight of the composition.
8. A composition as claimed in claim 6 or 7 wherein said non-ionic surfactant comprises fatty alcohol ethoxylate.
9. A composition as claimed in any one of the preceding claims comprising an alkaline agent selected from sodium carbonate or sodium hydroxide.

10. A composition as claimed in claim 9 wherein said alkaline agent is present in 0.1 to 1% by weight of the composition.
11. A method of cleaning and disinfecting a hard surface comprising applying to said hard surface a composition as claimed in any one of the preceding claims diluted with water in a ratio of 1:20 to 1:200.

## Patentansprüche

1. Flüssige antimikrobielle Reinigungszusammensetzung für harte Oberflächen mit einem pH-Wert in dem Bereich von 9 bis 12, umfassend
  - (a) 0,5 bis 4 Gew.-% Benzalkoniumchlorid;
  - (b) 0,25 bis 3 Gew.-% eines Salzes einer Di- oder Tricarbonsäure;
  - (c) 0,01 bis 2 Gew.-% eines antimikrobiellen Wirkstoffs aus essentiellm Öl, ausgewählt aus Thymol oder Terpeneol; und
  - (d) 87 bis 97 Gew.-% Wasser.
2. Zusammensetzung gemäß Anspruch 1, umfassend Thymol und Terpeneol.
3. Zusammensetzung gemäß Anspruch 1 oder 2, wobei das Salz einer Di- oder Tricarbonsäure aus einem Oxalat, Fumarat, Phthalat, Maleat, Malat, Malonat oder Citrat ausgewählt ist.
4. Zusammensetzung gemäß Anspruch 3, wobei das Salz einer Di- oder Tricarbonsäure aus einem Phthalat, Malat, Malonat oder Citrat ausgewählt ist.
5. Zusammensetzung gemäß irgendeinem der vorhergehenden Ansprüche, umfassend 0,25 bis 3 Gew.-% Natriumtripolyphosphat.
6. Zusammensetzung gemäß irgendeinem der vorhergehenden Ansprüche, umfassend ein nichtionisches Tensid.
7. Zusammensetzung gemäß Anspruch 6, wobei das nichtionische Tensid in einer Menge von 2 bis 10 Gew.-% der Zusammensetzung vorhanden ist.
8. Zusammensetzung gemäß Anspruch 6 oder 7, wobei das nichtionische Tensid Fettalkohol-Ethoxylat umfasst.
9. Zusammensetzung gemäß irgendeinem der vorhergehenden Ansprüche, umfassend ein alkalisches Mittel ausgewählt aus Natriumcarbonat oder Natriumhydroxid.
10. Zusammensetzung gemäß Anspruch 9, wobei das alkalische Mittel mit 0,1 bis 1 Gew.-% der Zusammensetzung vorhanden ist.
11. Verfahren zum Reinigen und Desinfizieren einer harten Oberfläche, umfassend das Aufbringen einer Zusammensetzung gemäß irgendeinem der vorhergehenden Ansprüche auf die harte Oberfläche, die in einem Verhältnis von 1:20 bis 1:200 mit Wasser verdünnt ist.

## Revendications

1. Composition nettoyante antimicrobienne liquide pour surface dure ayant un pH dans la plage de 9 à 12 comprenant
  - (a) de 0,5 à 4 % en poids de chlorure de benzalkonium ;
  - (b) de 0,25 à 3 % en poids de sel d'acide di- ou tricarboxylique ;
  - (c) de 0,01 à 2 % en poids d'une huile essentielle antimicrobienne active choisie parmi le thymol ou le terpinéol ; et
  - (d) de 87 à 97 % en poids d'eau.
2. Composition selon la revendication 1 comprenant du thymol et du terpinéol.
3. Composition selon la revendication 1 ou 2 dans laquelle ledit sel d'acide di- ou tricarboxylique est choisi parmi un

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oxalate, un fumarate, un phtalate, un maléate, un malate, un malonate ou un citrate.

- 5      **4.** Composition selon la revendication 3 dans laquelle ledit sel d'acide di- ou tricarboxylique acide est choisi parmi un phtalate, un malate, un malonate, ou un citrate.

- 10      **5.** Composition selon l'une quelconque des revendications précédentes comprenant 0,25 à 3 % de tripolyphosphate de sodium.

- 15      **6.** Composition selon l'une quelconque des revendications précédentes comprenant un tensioactif non ionique.

- 20      **7.** Composition selon la revendication 6 dans laquelle ledit tensioactif non ionique est présent en une quantité de 2 à 10 % en poids de la composition.

- 25      **8.** Composition selon la revendication 6 ou 7 dans laquelle ledit tensioactif non ionique comprend un éthoxylate d'alcool gras.

- 30      **9.** Composition selon l'une quelconque des revendications précédentes comprenant un agent alcalin choisi parmi le carbonate de sodium ou l'hydroxyde de sodium.

- 35      **10.** Composition selon la revendication 9 dans laquelle ledit agent alcalin est présent à 0,1 à 1 % en poids de la composition.

- 40      **11.** Procédé de nettoyage et de désinfection d'une surface dure comprenant l'application sur ladite surface dure d'une composition selon l'une quelconque des revendications précédentes diluée avec de l'eau dans un rapport de 1:20 à 1:200.

**REFERENCES CITED IN THE DESCRIPTION**

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