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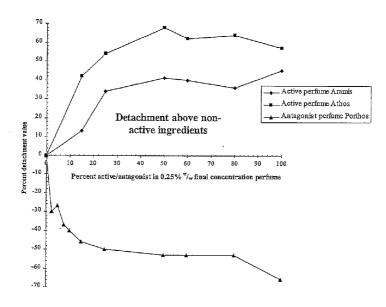
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#### (54) Title: PERFUME COMPOSITIONS



(57) Abstract: Perfume ingredients have been investigated and classified for their effectiveness in detaching biofilms from surfaces. Based on this classification, perfume compositions that are effective in assisting biofilm detachment can be formulated, whilst also allowing a degree of freedom in formulation that permits consideration of the hedonic properties of the compositions can be used in a wide range of perfumed consumer products, such as personal, domestic and industrial products, e.g. hard surface cleaning products, dental care products, and deodorant products.



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Title: Perfume Compositions

Field of the Invention

This invention relates to perfume compositions including flavour compositions. For the purposes of this invention a perfume composition is defined as a mixture of perfume ingredients, including if desired a suitable perfume solvent or solvents and optionally mixed with a solid substrate. Perfume ingredients are well known to those skilled in the art, and include those mentioned, for example, in S. Arctander, Perfume and Flavor Chemicals (Montclair, N.J., 1969), in S. Arctander, Perfume and Flavor Materials of Natural Origin (Elizabeth, N.J., 1960) and in "Flavor and Fragrance Materials – 1991", Allured Publishing Co. Wheaton, Ill. USA. Perfume ingredients may include natural products such as extracts, essential oils, absolutes, resinoids, resins, concretes etc., and also synthetic basic substances such as hydrocarbons, alcohols, aldehydes, ketones, ethers, acids, esters, acetals, ketals, nitriles, etc., including saturated and unsaturated compounds, aliphatic, arbocyclic and heterocyclic compounds. The invention is particularly concerned with perfume compositions that have the ability to detach or remove biofilms from surfaces.

Background to the Invention

Biofilms are collections of microorganisms (bacteria, fungi etc.) that are attached, directly or indirectly, to a solid phase abiotic or biotic surface. Biofilms grow in situ, and are usually embedded in extracellular polymeric substance (EPS) matrix of microbial or other origin. Biofilms are generally difficult to remove from a surface. The majority of bacteria in nature exist in biofilms, and biofilms often represent the "preferred" mode of growth for bacteria (Costerton JW, Lewandowski Z, Caldwell DE, Korber DR, Lappin-Scott HM (1995). Microbial biofilms. Annual Reviews of Microbiology Volume 49, pp. 711-745). Biofilms may be composed of only one type of microorganism, but frequently contain several types of microorganism living in a complex community. Biofilm communities can include bacteria, fungi, yeasts, protozoa, and other microorganisms. In

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fact, almost every surface exposed to liquids and nutrients can be colonised by microorganisms. Biofilms include those associated with animals (e.g. dental plaque in the mouth, bacteria on mucosal surfaces in the gut, or on the skin); pathogens that infect burns, wounds or implanted devices. Biofilms are also significant in a number of industrial settings, such as water and food processing systems. Biofilms are also a part of domestic life: in the home, biofilms are found in bathrooms, toilets, showers and kitchens. Biofilms are also of interest because they display a range of unusual properties – notably resistance to disinfection and removal. A great number of medical, industrial and domestic products aim to control biofilms, and many of these are fragranced.

Initially, the formation of a biofilm typically involves the adsorption of an organic monolayer ("conditioning film") to a surface that can change the chemical and physical properties of the surface. These adsorbed materials condition the surface and allow free-floating, pioneer microorganisms that encounter the conditioned surface to form a reversible, sometimes transient attachment. This attachment is influenced by electrical charges carried on the microorganism and by Van der Waals forces and by electrostatic attraction. Further chemical and physical structures can transform the reversible attachment to a permanent and essentially "irreversible" attachment, via adhesins on the surface of the microorganism and receptors on the surface. Following this irreversible attachment, the production of extracellular polymeric substance can allow entrapment of other materials (such as organic materials, dead cells and precipitated minerals etc.). These can add to the bulk and diversity of the biofilm. Pioneer attached bacteria may grow. Secondary colonizing bacteria may attach to the pioneer bacteria, and a mature biofilm community may develop.

Biofilms may be found on the surfaces of water tanks, pipes, surgical apparatus, food-processing vessels, etc., where the bacteria adhere tenaciously, resisting removal by washing and also gaining protection from antimicrobial compounds such as common disinfectants which cannot easily penetrate the polysaccharide matrix.

Biofilms can act as a source of infections. Examples include seeding water systems with *Legionella* bacteria, leading to Legionnaire's disease; in a medical setting, biofilms may be responsible for a range of life-threatening infections associated with burns or wounds, or with prosthetic devices such as heart valves, catheters and replacement joints; hospital equipment (e.g. artificial respirators and ventilators) can become colonized with bacteria, leading to infection of vulnerable patients.

A further example of a biofilm is the dental plaque found on teeth, which is the causative agent of dental caries when bacteria such as *Streptococcus mutans* degrade sugars to organic acids, or on oral mucosal surfaces where the dental plaque biofilm is the aetiological agent which can lead to the development periodontal (gum) diseases.

Biofilms can colonize many household surfaces, including toilets, sinks, countertops, and cutting boards in the kitchen and almost all of the various surfaces in bathrooms. These biofilms can act as a source of malodours, cause staining and may pose an infection hazard.

In industry, biofilms are responsible for enormous economic costs in lost industrial productivity and both product and capital equipment damage each year. For example, biofilms are notorious for causing pipe plugging, inefficiency of heat exchange systems, corrosion of metal surfaces and water contamination.

Biofilm contamination and fouling occurs in nearly every industrial water-based process, including food and drink production, water treatment and distribution, pulp and paper manufacturing, and the operation of cooling towers.

The most notable property of biofilms is their unusual resistance to a wide range of antimicrobial agents, which renders them difficult to disinfect. This inherent high resistance of biofilms is derived from a number of factors. The extracellular polymeric substance around biofilms may act as a barrier to diffusion of antimicrobials, as a food reserve and can prevent desiccation. Certain bacteria can secrete enzymes that inactivate

antimicrobials; other bacteria, growing in the close proximity that a biofilm allows, which otherwise would be sensitive to the antimicrobial, can themselves be rendered "resistant" when in a biofilm. Furthermore, evidence is growing that microorganisms in biofilms can display resistant phenotypes, resulting from altered patterns of gene expression and interbacterial communication. These potential mechanisms of biofilm resistance have been described in several recent reviews (for example see Gilbert P, Maira-Litran T, McBain AJ, Rickard AH, Whyte FW. (2002). The physiology and collective recalcitrance of microbial biofilm communities. *Advances in Microbial Physiology*. Volume 46, pp. 202-256).

Many products are designed, either directly or indirectly, to combat biofilms or biofilm-related problems. Such products generally involve some form of physical activity, optionally aided by cleaning materials such as surfactants, to detach the biofilm. Sometimes, these products also contain strong disinfectants, such as bleach or other antimicrobial actives, to kill or neutralize the microorganisms within the biofilm.

### Summary of the Invention

The present invention is based on the surprising discovery that certain perfume ingredients, individually or in combination, possess hitherto unknown biofilm-detachment properties. Such ingredients may be used to supplement the biofilm-detachment properties of a variety of products, for example general purpose cleaners.

We have carried out extensive testing of a large number of individual perfume ingredients and mixtures thereof to determine their effectiveness in detaching biofilms. This testing has enabled us to classify perfume ingredients into different categories, namely those that assist biofilm detachment (referred to herein as Group A materials), those that are non-active to biofilm detachment (referred to herein as Group B materials), and those that are detrimental or antagonistic to biofilm detachment (referred to herein as Group C materials). Complete detachment of biofilms is not essential, with partial removal of biofilms being sufficient. Based on this classification of perfume ingredients, the

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invention enables the formulation of perfume compositions that are effective in assisting biofilm detachment while allowing a degree of freedom in formulation that permits consideration of the hedonic properties of the composition. The invention can thus enable formulation of perfume compositions that are effective in detaching biofilms and that also have good hedonic properties which can be tailored to suit a range of different product types.

In particular, a standardised biofilm detachment assay was used to quantify perfume ingredients in biofilm removal in terms of percent detachment values (PDVs). standardised biofilm detachment assay is based on testing perfume compositions of 4 perfume ingredients present in equal amounts by weight, comprising 3 ingredients that fall in Group B and are non-active with regard to biofilm detachment (3,7-dimethyloct-6-en-1ol; 1-ethenyl-1,5-dimethylhex-4-enyl acetate; and phenylmethyl-2-hydroxybenzoate) in admixture of a perfume ingredient under test. Each 4 component perfume mixture was used at a concentration of 0.25% w/w in a quarter strength general purpose cleaner (GPC) base test mixture having the composition of Formula A as set out in Example 1(a) below. The test mixtures were tested by the procedure set out in Example 1(a) below to determine a PDV for the perfume composition under test, and hence a PDV of the particular 4th perfume ingredient under test in the mixture indicative of the biofilm removal efficacy of that ingredient. PDVs for the perfume ingredients obtained in this way were rounded to the nearest integer. References in this specification to percent detachment value or PDV of a material means a value obtained using this standardised biofilm detachment assay, as defined above.

Group A materials that assist biofilm detachment are defined as those having a PDV (as defined above) of at least 9. Group B materials that are non-active to biofilm detachment are defined as those having a PDV (as defined above) of less than 9 and greater than 0. Group C materials that are antagonistic to biofilm detachment are defined as those having a PDV (as defined above) of less than or equal to 0.

Examples of perfume ingredients falling into Groups A, B and C, are as follows:

patchouli oil §

phenylmethanol 5

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Group A (active) (PDV \geq 9)
     (2E)-tridec-2-enenitrile
    (2Z)-2-ethyl-4-(2,2,3-trimethylcyclopent-3-en-1-yl)but-2-en-1-ol
    (3E)-3-methyl-4-(2,6,6-trimethylcyclohex-2-en-1-yl)but-3-en-2-one
    1-(2,3,8,8-tetramethyl-1,2,3,5,6,7,8,8 a-octahydronaphthalen-2-yl) ethan one and the state of 
    1-(2.6.6.8-\text{tetramethyl-tricyclo}[5.3.1.0^{1.5}] undec-8-en-9-yl)ethanone
    1-(methyloxy)-4-[(1E)-prop-1-enyl]benzene <sup>¶</sup>
    1,4-dioxacycloheptadecane-5,17-dione
    1-[1,1,2,6-tetramethyl-3-(1-methylethyl)-2,3-dihydro-1H-inden-5-yl]ethanone
    1-methyl-4-(1-methylethyl)-2-[(1E)-prop-1-enyl]benzene
    1-methylethyl tetradecanoate
    2-(2-methylpropyl)-4-hydroxy-4-methyl-tetrahydropyran
    2,6,10-trimethylundec-9-enal
    2-[2-(4-methylcyclohex-3-en-1-yl)propyl]cyclopentanone
    2-methylundecanal
. 5-methyl-2-(1-methylethyl)phenol ¶
    anisic aldehyde
    cinnamic alcohol 9
    clove bud rectified extra DQ P353<sup>§</sup>
    cyclohexadecanolide
    cyclopentadecanone
    decanol DQ
    dodecanenitrile
    eucalyptol §
    eucalyptus globulus <sup>¶</sup>
    eugenol rectified<sup>§</sup>
    ginger oils <sup>¶</sup>
    isoamyl acetate
 ~nonanol <sup>¶</sup>
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propane-1,2-diol §
tea tree oil DO 1
tributyl-2-(acetyloxy)propane-1,2,3-tricarboxylate
1H-indole¶
(2E)-2-pentyl-3-phenylprop-2-enal
(4E)-4-tricyclo[5.2.1.0^{2,6}]dec-8-ylidenebutanal
2,6,6,8-tetramethyltricyclo[5.3.1.0^{1,5}]undec-8-yl acetate
2-[(2-{[2-(methyloxy)propyl]oxy}propyl)oxy]propan-1-ol
2-methyldecanenitrile
1-(1,1,2,3,3,6-hexamethyl-2,3-dihydro-1H-inden-5-yl)ethanone
1-(3,5,5,6,8,8-hexamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)ethanone
4-(1,7,7-trimethylbicyclo[2.2.1]hept-2-yl)cyclohexanol
Silvanone (Silvanone is a Trade Mark)
2-heptylcyclopentanone
3-methyldodecanenitrile
(6E)-3,7,11-trimethyldodeca-1,6,10-trien-3-ol
methyl (2E)-non-2-enoate
(2E)-2-hexyl-3-phenylprop-2-enal
ethyl 3-(1-methylethyl)bicyclo[2.2.1]hept-5-ene-2-carboxylate
3-methylcyclopentadecanone
ylang ylang oil
2-(1,1-dimethylethyl)cyclohexyl acetate
2,4-dimethyl-4,4a,5,9b-tetrahydroindeno[1,2-{d}][1,3]dioxine
4,6,6,7,8,8-hexamethyl-1,3,4,6,7,8-hexahydroindeno[5,6-{c}]pyran (e.g. in the
form of Galaxolide - Galaxolide is a Trade Mark)
[4-(1-methylethyl)cyclohexyl]methanol
capsicum oleoresin DQ
peppermint American lagrande DQ
lime terpeneless DQ
peppermint American yakima rectified DQ
citronella Ceylon DQ
peppermint arvensis co-distilled DQ
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rose flavour base ABF0339A
marjoram French DQ
jasmin absolute DQ
coriander DQ
cinnamic aldehyde extra DQ
citral
lavender oil DQ
iso propyl alcohol DQ
amyl cinnamic aldehyde DQ
amyl caproate DQ
peppermint Chinese triple rectified (Q) (DQ)

DQ = dental quality
Q = Quest International

ketones of general formula RCOR' having an octanol-water partition coefficient of at least 4 (expressed as a logarithm to base 10), where R and R' are independently hydrocarbyl residues that may be aliphatic or aromatic, saturated or unsaturated, and combinations thereof, but may not contain other functional groups.

The octanol-water partition coefficient (P) of a material i.e. the ratio of a material's equilibrium concentration in octanol and water, is well known in the literature as a measure of hydrophobicity and water solubility (see Hansch and Leo, Chemical Reviews, 526 to 616, (1971), 71; Hansch, Quinlan and Lawrence, J. Organic Chemistry, 347 to 350 (1968), 33). High partition coefficient values are more conveniently given in the form of their logarithm to the base 10, log P. While log P values can be measured experimentally i.e. directly, and measured log P data is available for many perfumes, log P values are most conveniently calculated or approximately estimated using mathematical algorithms. There are several recognised calculation or estimation methods available commercially and/or described in the literature (see for example A Leo, Chem. Rev 93(4), 1281-1306, (1993), "Calculating log P oct from structures"). Generally these models correlate highly but may for specific materials produce log P values which differ in absolute terms (by up

to 0.5 log units or even more). However, no one model is universally accepted as the most accurate across all compounds. This is particularly true for estimates on materials of high log P (say 4 or greater). In the present specification, log P values are obtained using the estimation software commercially available as 'Log P' from Toronto-based Advanced Chemistry Development Inc (ACD) which is well-known to the scientific community, and accepted as providing high-quality predictions of log P values. References to log P values thus mean values obtained using the ACD software.

Examples of suitable ketones having a log P (obtained using the ACD software) of at least 4 include the following:

1-(2,3,8,8-tetramethyl-1,2,3,4,5,6,7,8-octahydronaphthalen-2-yl)ethanone (log P=5.28)  $1-(2,6,6,8-\text{tetramethyltricyclo}[5.3.1.0^{1},5]\text{undec-}8-\text{en-}9-\text{yl})\text{ethanone (log P}=5.17)$  1-(1,1,2,3,3,6-hexamethyl-2,3-dihydro-1H-inden-5-yl)ethanone (log P=5.80) 1-(3,5,5,6,8,8-hexamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)ethanone (log P=6.37) 3-methylcyclopentadecanone (log P=6.33) 1-[1,1,2,6-tetramethyl-3-(1-methylethyl)-2,3-dihydro-1H-inden-5-yl]ethanone (log P=6.14)

<u>Group B</u> (non-active)(PDV > 0 and < 9)

(3Z)-hex-3-en-1-ol

1,7,7-trimethylbicyclo[2.2.1]hept-2-yl acetate

1-methyl-4-(1-methylethylidene)cyclohex-1-ene

2-(phenyloxy)ethanol

2,6,10-trimethyl-1-acetyl-cyclododeca-2,5,9-triene

2-{[2-(ethyloxy)ethyl]oxy}ethanol

3,7-dimethyloct-6-en-1-ol

diethyl benzene-1,2-dicarboxylate

(4E)-dec-4-enal

ethyl-2-methyl-1,3-dioxolan-2-yl acetate

5-heptyldihydrofuran-2(3H)-one<sup>§</sup>

origanum<sup>9</sup>

1-(methyloxy)propan-2-ol

2,2-dimethyl-3-(3-methylphenyl)propan-1-ol

tricyclo $[5.2.1.0^{2,6}]$ dec-4-en-8-yl propanoate

 $3a,6,6,9a-tetramethyl dode cahydron aphtho [2,1-\{b\}] fur an$ 

hexyl 2-hydroxybenzoate

phenylmethyl-2-hydroxybenzoate §

cyclohexadec-5-en-1-one

Rose fragrance 0409™ (available from Quest International)

1-ethenyl-1,5-dimethylhex-4-enyl acetate

(4Z)-dec-4-enal

1-(5,5-dimethylcyclohex-1-en-1-yl)pent-4-en-1-one

3-methyl-1-(2-methylpropyl)butyl acetate

2-(methyloxy)-4-propylphenol<sup>§</sup>

2-(acetyloxy)-1-[(acetyloxy)methyl]ethyl acetate

Thyme Red ¶

4-(1,1-dimethylethyl)cyclohexyl acetate

 $5\hbox{-methyl-}2\hbox{-}(1\hbox{-methylethyl})\hbox{cyclohexanol}$ 

Aldehyde C10 DQ<sup>¶</sup>

Lime Terpenes Washed DQ<sup>¶</sup>

Peppermint Piperita Indian Rectified FDC DQ

Anisic aldehyde

Blackcurrent Base ABF0972<sup>5</sup>

Peppermint Indian Rectified (FDC) DQ<sup>1</sup>

Grapefruit DQ

Parsley Herb<sup>§</sup>

Peppermint American Far West Rectified DQ<sup>5</sup>

Orange Terpenes Ex Concentrate DQ

Iso Amyl Butyrate DQ

Cinnamon Supra ABF1092<sup>f</sup>

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Cardamom English Distilled DQ

Apple Base ABF1016

Peppermint Chinese Terpeneless (Q) DQ<sup>f</sup>

Peppermint Moroccan DQ<sup>¶</sup>

Peppermint American Idaho DQ<sup>¶</sup>

Orange Terpeneless DQ

DQ = Dental Quality

O = Quest International

Group C (antagonist) (PDV  $\leq$  0)

2-methyl-3-[4-(methyoxy) phenyl] propanal

(2E)-3-phenylprop-2-enal

lemongrass oil

1,3-dimethylbut-3-enyl 2-methylpropanoate

Muguet base AB7001™ (Q)

Moss base AB7004™ (Q)

Sandalone AC802™ (Q)

Jasmin AB7002™ (Q)

prop-2-enyl [(2-methylbutyl)oxy]acetate

2,6-dimethylhept-5-en-2-ol

phenylmethyl acetate

Carvone

3-pentyltetrahydro-2H-pyran-4-yl acetate

3,7-dimethylocta-1,6-dien-3-ol

4-(4-hydroxy-4-methylpentyl)cyclohex-3-ene-1-carbaldehyde

methyl (3-oxo-2-pentylcyclopentyl)acetate

3-methyl-5-phenylpentan-1-ol

(1E)-1-(2,6,6-trimethylcyclohex-2-en-1-yl)pent-1-en-3-one

Orange oil

2-phenylethanol

2-(4-methylcyclohex-3-en-1-yl)propan-2-ol

1-[(2-hydroxypropyl)oxy]propan-2-ol

(6Z)-3,7-dimethylnona-1,6-dien-3-ol

Q = Materials available from Quest International

In the lists of Group A and Group B materials, those marked with the symbol ¶ are known to have some antimicrobial (bacteriostatic or bactericidal) properties. See, for example, the paper by Morris J A, Khettry A, Seitz E W (1979) Antimicrobial activity of aroma chemicals and essential oils. *Journal of the American Oil Chemistry Society* Volume 56, pp. 595-603; and WO 01/24769 of Firmenich & Cie.

In one aspect, the present invention provides a perfume composition comprising at least 15% by weight of at least 2 Group A perfume ingredients and wherein the Group A and Group B ingredients together constitute at least 80 wt % of the perfume composition, as defined above, i.e. having Group A and Group B being PDV values determined by the standardised biofilm detachment assay as defined herein.

The perfume composition preferably comprises at least 2, at least 3 or at least 4 Group A ingredients. The perfume composition may even comprise at least 5 or at least 6 Group A ingredients.

The use of a mixture of several Group A ingredients assists formulation of compositions having desired hedonic properties as well as biofilm detaching properties. The perfume composition may comprise at least 25% by weight, at least 30% by weight, at least 40% by weight or possibly at least 60% by weight of Group A perfume ingredients.

The perfume composition desirably includes at least one, preferably at least two or at least three or more Group B perfume ingredients, as defined above, i.e. having a PDV between 0 and 9 determined by the standardised biofilm detachment assay defined herein. These materials are not detrimental to the biofilm detaching properties of the composition and so

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enhance the scope for formulation of compositions having desired hedonic properties without having an adverse effect on biofilm detachment.

A preferred perfume composition comprises at least 3 Group A perfume ingredients and at least 3 Group B perfume ingredients.

The perfume composition should preferably not include any Group C perfume ingredients or should keep these to a minimum, as these will have an adverse effect on biofilm detachment properties of the composition. Group C ingredients are as defined above, i.e. having a PDV less than or equal to 0 determined by the standardised biofilm detachment assay defined herein. Perfume compositions in accordance with the invention comprise at most 20 wt % Group C ingredients. Preferably they comprise at most 15 wt %, at most 10 wt % or at most 5 wt % or even at most 3 wt % Group C ingredients.

Each of the Group A ingredients, and the Group B ingredients if present, is preferably present in an amount not exceeding 20% by weight, more preferably not exceeding 15% by weight and most preferably not exceeding 10% by weight.

Preferred ingredients of Groups A and B are those that deliver effective odour intensities in use, and are characterised by exhibiting odour threshold levels in air of lower than 2000 ppb (parts per billion) by weight, or even more preferably lower than 1000 ppb, and yet more preferably lower than 500 ppb. Odour thresholds are discussed and examples provided in many literature sources, among which may be mentioned in particular: "Standardized Human Olfactory Thresholds", M Devos et al, IRL Press at Oxford University Press (1990), and "Compilation of Odor and Taste Threshold Values Data", F A Fazzalari (editor), ASTM Data Series DS 48A (1978).

The perfume composition desirably includes one or more perfume ingredients with antimicrobial properties if such properties are desired, such as those materials identified by the symbol \( \frac{1}{2} \) in the lists above.

This invention concerns perfume compositions that have the ability to detach (i.e. interrupt and end the attachment of) biofilms that comprise, for example, mould, fungi, algae and/or bacteria, from surfaces and a process whereby an effective amount of the composition is applied to the surface and/or the biofilm(s) attached to the surface.

The biofilm-detaching perfume compositions of this invention desirably have the following characteristics:

- (1) do not leave a film under which microorganisms may remain viable.
- (2) are effective against a wide spectrum of microorganisms.
- (3) negate and retard the overgrowth of microorganisms.
- (4) prevent or retard biofilm formation.
- (5) may potentiate the biofilm detachment properties of bases and do not retard base activity.

The mode of action of this invention is not systemic. The mode of action is universal in nature, so that the overgrowth of any microbial species may be prevented.

The compositions of the invention are effective against biofilms on all types of solid or semi-solid surfaces, as can be found e.g. in a household such as, glass, stone, wood, metal, plastic, fabrics, gauze, ceramic, porcelain and animal or human tissue (e.g. a skin surface or an oral surface), vegetable matter and the like.

The compositions of this invention are effective against microorganisms, such as, mould, fungi, algae, yeast, bacteria (Gram-negative and Gram-positive) and viruses.

The modes of action of the biofilm-detaching compositions of this invention are not necessarily bacteriostatic or bactericidal or antibacterial or the like, in the normal sense of such terms. Instead, the compositions detach microorganisms and bring about a reduction in the populations of microorganisms on the surface. However, the compositions of the invention can be so designed, if required, to deliver explicit antimicrobial effects (e.g.

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bacteriostatic, bactericidal etc), in addition to the biofilm removing effect. Alternatively, compositions with low or no inherent antimicrobial activity may also be produced.

The perfume compositions of the present invention may be incorporated into a wide variety of consumer products, particularly personal, domestic and industrial products, to provide not only desirable odour properties but also to deliver or enhance the biofilm-detachment properties of such products. The consumer products may be in a range of the different physical forms, including slurries, suspensions, solutions, pastes, emulsions, etc.

In particular, the compositions of the invention may find application in all types of cleaning products (household, medical, industrial), particularly hard surface cleaning For example, they could be used in general-purpose-cleaners or all purpose cleaners, dishwashing products (either hand- or machine-dishwashing), or cleaners for kitchen surfaces such as sinks, worktops, ovens, utensils; cleaners for all types of domestic floors such as tiles, wood, carpets, cement and linoleum; they could be useful in cleaners for domestic appliances such as washing machines, dishwashers, food processors, vacuum cleaners and the like; in the bathroom they could find application in cleaners for showers, baths, taps, plugs, drains, toilets, bidets, washbasins, surrounding tiles, shower curtains and doors; for toilet products they could also find application in under-the-rim products (rim liquids, rim blocks), in-tank products (blocks and liquids), on-tank products; in the wider domestic environment they may find application in car, shoe/boot, window, wall, ceiling and other miscellaneous cleaning products; they may be applied in a range of household polishing products; outside the home they may find application in cleaners or treatments for concrete, tarmac, pipelines, wooden surfaces, brickwork, garden furniture, domestic ponds, alloy wheels and metals, glass, fountains and the like. In medical and industrial settings they may find application in a wide variety of cleaning products with possibly different product delivery mechanisms in the form of fragrance cartridges, multinozzle dispensing containers etc.

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Additionally they may find application in a range of personal care, hair care, skin care and cosmetic products. For example, they may be used in face creams, lipsticks, makeup/nail removers, sun creams, hand and body washing products. The composition of this invention can be used as a deodorant. It can be directly applied as a liquid, or can be placed on an absorbent pad or the like and applied in liquid form by putting on the affected surface. Adhesion of microbes, that directly or indirectly cause body odours, is prevented or retarded, and such microbes are detached. The compositions and methods of this invention may be useful also in the treatment of dermatitis and itching caused by microbes, to coat first-aid products (e.g., gauze pads, band-aids, etc.), to treat athlete's feet, in cosmetic preparation (e.g., base lotion, eye mascara, etc.), for washing food and meat cases, food counters and the like, to treat cuts, scratches and the like, as an eye wash, to treat dandruff, treatment of acne, to clean contact lenses, shaving creams, hand creams, and after shave lotions, ear wax removal, in aerosol sprays, in a liquid dentifrice, as a disinfectant and in shampoos.

Furthermore, the compositions of the invention can find application in dental care products such as dentifrices, denture care products, mouthwashes, breath freshening powders/tablets, breath films, teeth whitening products, chewing gums (where the term "chewing gum" is intended also to encompass bubble gum), dental floss, dissolvable mouth films, lozenges, gels, mousse, creams, aerosols (breath sprays) etc. to assist in the removal of dental plaque, and contribute in this way to dental hygiene. The compositions of the invention may also be used in oral care products. Also included are over the counter or prescription products for the prevention or treatment of gingivitis, plaque, tartar, caries and oral malodour.

The appropriate effective dosage of perfume composition in each product will depend upon the nature and purpose of the product. For example, in a window cleaner product the minimum useful dosage of a perfume composition of the invention is 0.05% by weight, more preferably 0.1% or 0.2% by weight, whereas in a rim-block toilet product the dosage would be much higher, around 5% or 10% by weight, or even higher. Suitable

levels of perfume composition in different product types can be readily determined, and are known to those skilled in the art.

In a further aspect, the invention provides a perfumed product comprising a perfume composition in accordance with the invention.

The invention also covers use of a perfume ingredient in Group A for the purpose of detaching a biofilm. Preferred features of this aspect are as discussed above in connection with the perfume composition of the invention.

The perfume composition is conveniently present in a consumer product, e.g. as discussed above. The composition or product can be readily applied to a surface in known manner, appropriate to the form and type of the composition or product. Appropriate dosages of composition or product and treatment times can be readily determined by one skilled in the art.

Also within the scope of the invention is a method of detaching a biofilm from a surface, comprising application to the surface of a perfume composition or perfumed product in accordance with the invention. The treatment can be repeated as required, for long term effectiveness.

The invention will be further described, by way of illustration, in the following Examples and with reference to the accompanying Figures, in which:

Figure 1 is a graph of percent detachment value (PDV) versus percent active (Group A)/antagonist (Group C) material in perfume compositions as tested in Example 5;

Figure 2 is a graph of percent detachment value (PDV) versus percent antagonist (Group C) material in perfume compositions as tested in Example 6; and

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Figure 3 is a bar chart of percent detachment value (PDV) for perfume compositions in water as tested in Example 7.

Examples

Biofilm-Detachment and Kill Assays Example 1

Example 1(a): Biofilm Detachment

A bacterial culture of the micro-organism Escherichia coli ATCC 10536 (American Type Culture Collection (ATCC), P.O. Box 1549, Manassas, VA 20108, USA) was grown overnight at 37°C in Tryptone Soya Broth (TSB) (Oxoid, Basingstoke, UK). The E. coli suspension was diluted a thousand fold in TSB and 150 µl of this dilution was then added to each well in a 96 well microtitre plate (Sterilin, Staffs, UK). The microtitre plate lids were then tape-sealed and incubated at 37°C, static. After 18 hours incubation, the bacterial suspension was discarded from each well followed by the addition of 200  $\,\mu l$ sterile de-ionised water. This step represented a wash cycle and a total of two of these wash cycles were conducted. In this way, test biofilms remaining attached were produced within the wells.

Perfume compositions comprising perfume ingredients to be tested were prepared and the compositions added to a high impact general purpose cleaner base (diluted to quarter strength) together with de-ionised water and the surfactant Synperonic 91/10 (Synperonic is a Trade Mark) to produce test mixtures with the composition as set out in Formula A below. (Synperonic 91/10 is a mixture of ethoxylated alcohols averaging C9-C11 (10 EOs)) that functions as a solubiliser for the perfume ingredients to aid fragrance solubility.

Formula A:

0.25% w/w perfume composition

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0.5% w/w Synperonic 91/10 (85% w/w) (Uniqema, Wirral, UK)

2.25%w/w de-ionised water

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97%w/w High Impact GPC base diluted with water to quarter strength, the composition of which (after dilution to quarter strength) is as follows:

1% w/w Texapon NSO/IS

0.25% w/w Ethanol (96%)

0.05% w/w Bronidox L

95.7% w/w Water (demineralised)

Texapon NSO/IS comprises sodium lauryl ether sulphate (28%) and is available from Henkel, Hertfordshire, UK (Texapon is a Trade Mark). Bronidox L comprises 5-bromo-5-nitro-1.3 dioxane available from Henkel, Hertfordshire, UK (Bronidox is a Trade Mark).

200  $\mu$ l of each test mixture was added to a total of eight wells, representing a column in the microtitre plate. Controls included a) formula A without perfume and with additional water in place of the perfume composition, and b) sodium lauryl sulphate (SLS) (2%) final concentration prepared in deionised water). Controls were added to separate microtitre plate columns. SLS is known to have biofilm detachment properties and is used as a positive control to provide an indication of the maximum level of detachment that could be obtained. Water alone was added to a single column to act as a negative control. All of the test mixtures and controls were left in the wells for 20 minutes, static, at 21° C. The contents of each well were then discarded and two wash cycles conducted. Next,  $200\mu$ l of crystal violet stain solution (0.01%) was added and left static for 15 minutes at 21°C. Again the contents of each well were discarded and two wash cycles conducted. Finally, the crystal violet was solubilised by adding  $200\mu$ l ethanol (70%) to each well for 15 minutes at 21°C, static.

The crystal violet binds to any remaining biofilm, and is used to quantify the amount of biofilm remaining attached in the wells, and hence the amount of biofilm removal. The method used was based on the technique described in the paper by O'Toole G.A., Pratt L.

A., Watnick P. I., Newman D. K., Weaver V. B., Kolter R. (1999) Genetic approaches to the study of biofilms. Methods in Enzymology, Vol. 310, pp. 91-109.

The crystal violet was quantified by measuring the optical density at 540 nm  $(OD_{540})$  of each well using a plate reader (model MRX, Dynatech laboratories, Chantilly, VA, USA). A percentage detachment value (PDV) for each test mixture was then calculated using the following formula:

PDV = 100-([OD<sub>540</sub> for Test Mixture  $\div$  OD<sub>540</sub> for Water Control] x 100).

Experiments showed that a test mixture comprising the ¼ strength base, Synperonic 91/10 and water but no perfume composition gave a maximum percentage detachment value of 20%. This was thus deducted from the PDV value determined as set at above to give Detachment Index (DI) for each perfume composition tested. The SLS positive control gave a PDV of about 80%.

Using the procedure set out in Example 1(a), individual perfume ingredients and also perfume compositions containing mixtures of perfume ingredients prepared in quarter strength GPC base were investigated and the biofilm detaching properties of perfume ingredients and mixtures were determined. Using these results individual perfume ingredients falling into Groups A, B and C defined above were identified. Based on the results of these experiments, perfume compositions comprising mixtures of perfume ingredients with potentially good biofilm detaching properties were formulated and formulation ingredient studies were carried out. Test formulations were then further tested using the procedure of Example 1(b).

# Example 1(b) Biofilm detachment - in-use simulation study

White ceramic tiles 38mm x 38mm were cleaned by submerging in ethanol (70%) white followed by a wash in de-ionised water and then towel dried. A culture of the microorganism *Escherichia coli* ATCC 10536 was grown overnight at 37°C in (TSB). The E.

coli suspension was diluted a thousand fold in TSB and tiles were submerged in the bacterial suspension and incubated at 37°C, static.

After 18 hours incubation, the tiles were removed and placed in a series of glass Petri dishes with the test surface of each tile face up. A total of five tiles were added to each Petri dish. One Petri dish was prepared in this way per test ingredient or control. 100ml of test ingredients or compositions were added to each Petri dish and maintained at 24°C, at 60 revolutions per minute for 20 minutes in a shaker incubator (model 4628-1GMNPCE, Jencons, Bedfordshire, UK). In addition, controls included were sodium lauryl sulphate (0.28% //w), water, formula A without perfume and air only. After 20 minutes exposure, the test ingredient was discarded and the tiles washed in de-ionised water. Crystal violet (0.01% //w final concentration) was then added to the tiles for five minutes at 21°C, static.

The tiles were then removed from the stain and washed in de-ionised water. The tiles were then allowed to dry and digital photographs were taken of the tiles. In addition, visual judgment using a score system of 0-5 was used to compare the degree of biofilm removal by the ingredients. A score of 0 represented a completely clean tile and 5 a tile covered with untreated biofilm as a control.

These investigations demonstrated that this invention can deliver measurable differences in activity on biofilm-soiled surfaces, and clearly show active ingredients that enhance the cleaning and/or disinfection of biofilms for hygiene, cleaning and oral care products.

# Example 2

# Biofilm-Detaching Composition A

A biofilm-detaching perfume composition in accordance with the present invention was prepared by mixing equal concentrations of six active (Group A) ingredients as listed below:

1-(methyloxy)-4-[(1E)-prop-1-enyl]benzene

1-[1,1,2,6-tetramethyl-3-(1-methylethyl)-2,3-dihydro-1H-inden-5-yl]ethanone

Dodecanenitrile

(2E)-tridec-2-enenitrile

1-(2,6,6,8-tetramethyltricyclo $[5.3.1.0^{1,5}]$ undec-8-en-9-yl)ethanone

1-methyl-4-(1-methylethyl)-2-[(1E)-prop-1-enyl]benzene

The perfume composition was used at a concentration of 0.25% w/w in the quarter strength GPC base composition of Formula A above, and was tested by the procedures of Examples 1(a) and 1(b). This composition gave a mean DI value of 28% (for tests of 4 samples, i.e. n=4) determined using the procedure set out in Example 1(a).

### Biofilm-Detaching Composition B

A biofilm-detaching perfume composition in accordance with the present invention was prepared by mixing equal concentrations of six active (Group A) ingredients as listed below:

1-(methyloxy)-4-[(1E)-prop-1-enyl]benzene

Dodecanenitrile

1-(2,6,6,8-tetramethyltricyclo[5.3.1.0 $^{1,5}$ ]undec-8-en-9-yl)ethanone

1-methylethyl tetradecanoate

2-methylundecanal

1,4-dioxacycloheptadecane-5,17-dione

The perfume composition was used at a concentration of 0.25% w/w in the quarter strength GPC base composition of Formula A above, and was tested by the procedures of Examples 1(a) and 1(b). This composition gave a mean DI value of 33% (n=4) determined using the procedure set out in Example 1(a).

## Biofilm-Detaching Composition C

A biofilm-detaching perfume composition in accordance with the present invention was prepared by mixing equal concentrations of six active (Group A) ingredients as listed below:

- $1\hbox{-}[1,1,2,6\hbox{-tetramethyl-}3\hbox{-}(1\hbox{-methylethyl})\hbox{-}2,3\hbox{-dihydro-}1H\hbox{-inden-}5\hbox{-yl}] e than one$
- (2E)-tridec-2-enenitrile
- 1-methyl-4-(1-methylethyl)-2-[(1E)-prop-1-enyl]benzene
- (2Z)-2-ethyl-4-(2,2,3-trimethylcyclopent-3-en-1-yl)but-2-en-1-ol
- (3E)-3-methyl-4-(2,6,6-trimethylcyclohex-2-en-1-yl)but-3-en-2-one
- 1-(2,3,8,8-tetramethyl-1,2,3,5,6,7,8,8a-octahydronaphthalen-2-yl)ethanone

The perfume composition was used at a concentration of 0.25% w/w in the quarter strength GPC base composition of Formula A above, and was tested by the procedures of Examples 1(a) and 1(b). This composition gave a mean DI value of 26% (n=4) determined using the procedure set out in Example 1(a).

# Biofilm-Detaching Composition D

A biofilm-detaching composition in accordance with the present invention was prepared by mixing equal concentrations of four active (Group A) ingredients and two non-active (Group B) ingredients as listed below:

- 1-(methyloxy)-4-[(1E)-prop-1-enyl]benzene\*
- 1-[1,1,2,6-tetramethyl-3-(1-methylethyl)-2,3-dihydro-1H-inden-5-yl]ethanone\*

Dodecanenitrile\*

- (2E)-tridec-2-enenitrile\*
- 2-(phenyloxy)ethanol \*\*.
- 2,2-dimethyl-3-(3-methylphenyl)propan-1-ol\*\*

\*Group A

\*\*Group B

The perfume composition was used at a concentration of 0.25% w/w in the quarter strength GPC base composition of Formula A above, and was tested by the procedures of Examples 1(a) and 1(b). This composition gave a mean DI value of 19% (n=2) determined using the procedure set out in Example 1(a).

## Example 3

The procedure described in example 1(a) was repeated but wherein flavour compositions comprising flavour ingredients from the oral care palette to be tested were prepared. The compositions were added to a modified transparent silica toothpaste base (diluted to quarter strength) together with de-ionised water to produce test mixtures with the composition as set out in Formula B below.

#### Formula B:

0.25 % w/w flavour composition

99.75%w/w Modified transparent silica toothpaste base diluted with water to quarter strength, the composition of which (after dilution to quarter strength) is as follows:

16.25% w/w Sorbitol (70% $^{\text{w}}$ / $_{\text{w}}$ ) Neosorb $^{\text{@}}$ 

0.062% w/w sodium fluoride

0.025% w/w Tri sodium phosphate anhydrous

1.0% w/w PEG 1500

0.2% w/w Saccharin (25% w/w solution prepared in de-ionised water)

to 100% w/w Water (demineralised)

Sorbitol is available from Roquette, France. Tri sodium phosphate anhydrous is available from ThermPhos, Oldbury, UK. Polyethylene glycol (PEG) is available from Univar,

Cheshire, UK. Saccharin is available from Ellis and Everard, UK. Sodium fluoride is available from Sigma, Dorset, UK.

A percentage detachment value (PDV) for each test mixture was then calculated using the following formula:

PDV = 100-([OD<sub>540</sub> for Test Mixture  $\div$  OD<sub>540</sub> for Water Control] x 100).

Experiments showed that a test mixture comprising the ¼ strength modified transparent silica toothpaste base but no flavour composition gave a percentage detachment value of approximately -10%. The PDV scores were therefore adjusted to account for base influences on flavour behaviour and Detachment Index (DI) values quoted for each flavour composition. The SLS positive control gave a PDV of about 80%.

Individual flavour ingredients and also flavour compositions containing mixtures of flavour ingredients prepared in quarter strength modified transparent silica toothpaste base were investigated and the biofilm detaching properties of flavour ingredients and mixtures were determined. Using these results individual flavour ingredients falling into Groups A, B and C defined above were identified. Based on the results of these experiments, flavour compositions comprising mixtures of flavour ingredients with potentially good biofilm detaching properties were formulated and formulation ingredient studies were carried out.

# Biofilm detaching composition E

A biofilm-detaching flavour composition in accordance with the present invention was prepared by mixing equal concentrations of four active (Group A), one neutral (Group B) and one antagonist (Group C) ingredients as listed below:

Lavender oil\*

Tea tree oil\*

Peppermint arvensis co-distilled\*

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Coriander\*

Origanum\*\*

Lemongrass oil\*\*\*

\* Group A

\*\* Group B

\*\*\* Group C

The flavour composition was used at a concentration of 0.25% w/w in the quarter strength modified transparent silica toothpaste of Formula B above, and was tested by the procedure of Example 1 (a). This composition gave a mean DI value of 31% (for tests of 4 samples, i.e. n=4) determined using the procedure set out in Example 1(a).

## Biofilm detaching composition F

A biofilm-detaching flavour composition in accordance with the present invention was prepared by mixing equal concentrations of six active (Group A) ingredients as listed below:

Capsicum oleoresion

Lime terpeneless

Peppermint American Lagrande

Citronella Ceylon

Clove bud rectified Extra

Marjoram French

The flavour composition was used at a concentration of 0.25% w/w in the quarter strength modified transparent silica toothpaste of Formula B above, and was tested by the procedure of Example 1 (a). This composition gave a mean DI value of 56% (for tests of 4 samples, i.e. n=4) determined using the procedure set out in Example 1(a).

## Biofilm detaching composition G

A biofilm-detaching flavour composition in accordance with the present invention was prepared by mixing equal concentrations of five active (Group A) ingredients and one non-active (Group B) ingredients as listed below:

Capsicum oleoresion\*

Lime terpeneless\*

Lavender oil\*

Tea tree oil\*

Anethole synthetic\*

Blackcurrent base ABF 0972\*\*

\* Group A

\*\* Group B

The flavour composition was used at a concentration of 0.25% w/w in the quarter strength modified transparent silica toothpaste of Formula B above, and was tested by the procedure of Example 1 (a). This composition gave a mean DI value of 43% (for tests of 4 samples, i.e. n=4) determined using the procedure set out in Example 1(a).

### Example 4

A biofilm-detaching composition in accordance with the present invention having both biofilm detachment and deodorant odour-reducing properties was prepared, having the following composition:

Group	Ingredient	% by weight (total ingredients $100\%$ $^{\text{w}}/_{\text{w}}$ )
В	phenylmethyl 2-hydroxybenzoate	25
A	(3Z)-hex-3-enyl 2-hydroxybenzoate	6
В	2-(methyloxy)-4-propylphenol	4
A	1,4-dioxacycloheptadecane-5,17-dione	6

A	1,4-dioxacycloheptadecane-5,17-dione	6
A	4-methyl-2-(2-methylpropyl)tetrahydro-	22
D	2H-pyran-4-ol Geranium African Blk 0901	2
D	2-hexylcyclopent-2-en-1-one	2
À	1-methylethyl tetradecanoate	25.6
В	Raspberry ketone 10% CARB AA 2422	4
D	Sage clary	2.4
A	1-(2,6,6,8- tetramethyltricyclo[5.3.1.0^{1,5}]undec -8-en-9-yl)ethanone	1
	Percent detachment value (PDV)	18
	5 hour % odour reduction score	22

D indicates that the material is not yet identified, but is tentatively allocated to group A or B.

The perfume composition was used at a final concentration of 1% w/w in the quarter strength GPC base composition of Formula A, and was tested by procedure Example 1(a) for biofilm detachment score. The result was adjusted to give a PDV value of 0 for the unperfumed composition.

The perfume composition embodying this invention was made and tested for deodorant action in an underarm product, using the deodorant value test generally as described in US-4-289641 using the formulation described in Formulation 1. The deo composition and the method for the deodorant value test are set out below.

#### Formulation 1:

Ingredient	Content (% by weight)		
Quest fragrance	1.0		
Isopropyl myristate	1.0		
Propellant* 40 psig	60.0		
Ethanol B	to 100.00		

\*Hydrocarbon propellant. This can be any deodorised blend of n-propane, n-butane or isobutane having a pressure of 40 pounds per square inch gauge or 2.812kg/cm<sup>2</sup> gauge (337 kPa).

The deodorant value test was carried out using a panel of Caucasian male subjects. A standard quantity (2 second spray) of an aerosol product containing the perfume composition or a soap control was applied to the axillae of the panel members in accordance with a statistical design.

Average scores for each test product and the control product were then determined and the score for each test product was subtracted from the score for the control product. A percent odour reduction score was determined for the test deo perfume.

### Example 5

Perfume compositions containing a total of up to 15 ingredients at differing concentrations were selected from the active (Group A), non-active (Group B) and antagonist (Group C) ingredient list and formulated in the quarter strength GPC base composition of Formula A above. A non-active perfume composition contained 11 Group B ingredients. Two active perfume compositions called Aramis (Table 1) and Athos (Table 2) each additionally contained 4 Group A ingredients. An antagonist perfume composition called Porthos (Table 3) additionally contained 4 Group C ingredients. The final concentration of the perfume compositions as tested was 0.25% w/w in GPC (Formula A), consistent with previous investigations. The percent active or antagonist ingredient in each perfume composition ranged from 0 to 100 percent with the remainder of the formulation containing non-active (Group B) perfume ingredients. The perfume compositions were tested using the microtitre plate technique as set out in Example 1(a) and a percent detachment value (PDV) calculated for each perfume composition. The results were adjusted to give a PDV value of 0 for the non-active perfume composition, containing Group B ingredients only. These results are presented in Tables 1 to 3 and in Figure 1 for the perfume formulations as listed below. In addition, a typical household cleaning product perfume (the composition of which is set out in Table 4) was also tested in the same way for biofilm detachment properties at a final concentration of 0.25% w/w in the quarter strength GPC base of formula A.

Table 1 Active perfume composition Aramis

	T	% by weight (total ingredients $100\%$ "/w)							
Group	Ingredient		15	25	50	60	80	100	
В	Rose fragrance 0409™ (available from Quest International)	11.1	9.4	8.3	5.6	4.4	2.2	0	
В	Ethyl-2-methyl-1,3-dioxolan-2-yl acetate	5.6	4.7	4.2	2.8	2.2	1.1	0	
В	5-heptyldihydrofuran-2(3H)- one	2.8	2.4	2.1	1.4	1.1	0.6	0	
В	Origanum	13.9	11.8	10.4	6.9	5.6	2.8	0	
В	1-(methyloxy)propan-2-ol	13.9	11.8	10.4	6.9	5.6	2.8	0	
В	2,2-dimethyl-3-(3-methylphenyl)propan-1-ol	8.3	7.1	6.3	4.2	3.3	1.7	0	
В	tricyclo[5.2.1.0^{2,6}]dec-4-en-8-yl propanoate	13.9	11.8	10.4	6.9	5.6	2.8	0	
В	1,7,7- Trimethylbicyclo[2.2.1]hept- 2-yl acetate	5.6	4.7	4.2	2.8	2.2	1.1	0	
В	2-{[2- (Ethyloxy)ethyl]oxy}ethanol	13.9	11.8	10.4	6.9	5.6	2.8	0	
В	3,7-Dimethyloct-6-en-1-ol	8.3	7.1	6.3	4.2	3.3	1.7	0	
В	3a,6,6,9a- tetramethyldodecahydronapht ho[2,1-{b}]furan	2.8	2.4	2.1	1.4	1.1	0.6	0	
A	1-Methyl-4-(1-methylethyl)- 2-[(1E)-prop-1-enyl]benzene	0.0	5.4	8.9	17.9	21.4	28.6	35.7	
A	3-methylcyclopentadecanone	0.0	4.3	7:.1	14.3	17.1	22.9	28.6	
. A	Ethyl 3- (1methylethyl)bicyclo[2.2.1] hept-5-ene-2-carboxylate	0.0	3.2	5.4	10.7	12.9	17.1	21.4	
A	Ylang Ylang oil	0.0	2.1	3.6	7.1	8.6	11.4	14.3	
	Percent detachment value (PDV)	0	13	34	41	40	36	45	

Table 2 Active perfume composition Athos

	T Y	% by weight (total ingredients 100%					'/ <sub>w</sub> )	
Group	Ingredient	0	15	25	50	60	80	100
В	Rose fragrance 0409™ (available from Quest International)	11.1	9.4	8.3	5.6	4.4	2.2	0
В	Ethyl-2-methyl-1,3- dioxolan-2-yl acetate	5.6	4.7	4.2	2.8	2.2	1.1	0
В	5-heptyldihydrofuran- 2(3H)-one	2.8	2.4	2.1	1.4	1.1	0.6	0
В	Origanum	13.9	11.8	10.4	6.9	5.6	2.8	0
В	1-(methyloxy)propan-2- ol	13.9	11.8	10.4	6.9	5.6	2.8	0
В	2,2-dimethyl-3-(3-methylphenyl)propan -1-ol	8.3	7.1	6.3	4.2	3.3	1.7	0
В	tricyclo[5.2.1.0^{2,6}]d ec-4-en-8-yl propanoate	13.9	11.8	10.4	6.9	5.6	2.8	0
В	1,7,7- Trimethylbicyclo[2.2.1]h ept-2-yl acetate	5.6	4.7	4.2	2.8	2.2	1.1	0
В	2-{[2- (Ethyloxy)ethyl]oxy}etha nol	13.9	11.8	10.4	6.9	5.6	2.8	0
В	3,7-Dimethyloct-6-en-1-ol	8.3	7.1	6.3	4.2	3.3	1.7	0
В	3a,6,6,9a- tetramethyldodecahydron aphtho[2,1-{b}]furan	2.8	2.4	2.1	1.4	1.1	0.6	0
A	(2E)-tridec-2-enenitrile	0.0	5.4	8.9	17.9	21.4	28.6	35.7
A	1-(2,6,6,8- tetramethyltricyclo[5.3.1 .0^{1,5}]undec-8-en-9- yl)ethanone	0.0	4.3	7.1	14.3	17.1	22.9	28.6
A	1-methylethyl tetradecanoate	0.0	3.2	5.4	10.7	12.9	17.1	21.4
A	(2Z)-2-ethyl-4-(2,2,3-trimethylcyclopent-3-en-1-yl)but-2-en-1-ol	0.0	2.1	3.6	7.1	8.6	11.4	14.3
	Percent detachment value (PDV)	0	42	54	68	62	64	57

Table 3 Antagonist perfume composition Porthos:

C	Tu and Jiant	% by weight (total ingredients 100% w/w)							
Group	Ingredient	0	2.5	5	7.5	10	15	25	
В	Rose fragrance 0409™ (available from Quest International)	11.1	10.8	10.6	10.3	10.0	9.4	8.3	
В	Ethyl-2-methyl-1,3- dioxolan-2-yl acetate	5.6	5.4	5.3	5.1	5.0	4.7	4.2	
В	5-heptyldihydrofuran- 2(3H)-one	2.8	2.7	2.6	2.6	2.5	2.4	2.1	
В	Origanum	13.9	13.5	13.2	12.8	12.5	11.8	10.4	
В	1-(methyloxy)propan-2- ol	13.9	13.5	13.2	12.8	12.5	11.8	10.4	
В	2,2-dimethyl-3-(3- methylphenyl)propan-1- ol	8.3	8.1	7.9	7.7	7.5	7.1	6.3	
В	tricyclo[5.2.1.0^{2,6}]d ec-4-en-8-yl propanoate	13.9	13.5	13.2	12.8	12.5	11.8	10.4	
В	1,7,7- Trimethylbicyclo[2.2.1]h ept-2-yl acetate	5.6	5.4	5.3	5.1	5.0	4.7	4.2	
В	2-{[2- (Ethyloxy)ethyl]oxy}etha nol	13.9	13.5	13.2	12.8	12.5	11.8	10.4	
В	3,7-Dimethyloct-6-en-1-ol	8.3	8.1	7.9	7.7	7.5	7.1	6.3	
В	3a,6,6,9a- tetramethyldodecahydron aphtho[2,1-{b}]furan	2.8	2.7	2.6	2.6	2.5	2.4	2.1	
С	2-methyl-3-[4- (methyoxy) phenyl] propanal	0.0	0.9	1.8	2.7	3.6	5.4	8.9	
С	(2E)-3-phenylprop-2- enal	0.0	0.7	1.4	2.1	2.9	4.3	7.1	
C	Lemongrass oil	0.0	0.5	1.1	1.6	2.1	3.2	5.4	
С	1,3-dimethylbut-3-enyl 2-methylpropanoate	0.0	0.4	0.7	1.1	1.4	2.1	3.6	
	Percent detachment value (PDV)	0	-30	-27	-37	-40	-46	-50	

Table 3 (continued)

		% by weight (total					
Group	Ingredient	ingredients 100% <sup>w</sup> / <sub>w</sub> )					
		50	60	.80	100		
В	Rose fragrance 0409™ (available from Quest International)	5.6	4.4	2.2	0		
В	Ethyl-2-methyl-1,3- dioxolan-2-yl acetate	2.8	2.2	1.1	0		
В	5-heptyldihydrofuran- 2(3H)-one	1.4	1.1	0.6	0		
В	Origanum	6.9	5.6	2.8	0		
В	1-(methyloxy)propan- 2-ol	6.9	5.6	2.8	0		
В	2,2-dimethyl-3-(3-methylphenyl)propan- 1-ol	4.2	3.3	1.7	0		
В	tricyclo[5.2.1.0^{2,6}] dec-4-en-8-yl propanoate	6.9	5.6	2.8	0		
В .	1,7,7- Trimethylbicyclo[2.2.1]hept-2-yl acetate	2,8	2.2	1.1	0		
В	2-{[2- (Ethyloxy)ethyl]oxy}et hanol	6.9	. 5.6	2.8	0		
В	3,7-Dimethyloct-6-en- 1-ol	4.2	3.3	1.7	0		
В	3a,6,6,9a- tetramethyldodecahydr onaphtho[2,1-{b}]furan	1.4	1.1	0.6	0		
С	2-methyl-3-[4- (methyoxy) phenyl] propanal	17.9	21.4	28.6	35.7		
С	(2E)-3-phenylprop-2-enal	14.3	17.1	22.9	28.6		
C	Lemongrass oil .	10.7	12.9	17.1	21.4		
С	1,3-dimethylbut-3-enyl 2-methylpropanoate	7.1	8.6	11.4	14.3		
	Percent detachment value (PDV)	-53	-53	-53	-66		

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These results are summarised in Figure 1. A plateau response can be observed at  $\geq 25\%$  w/w active (Group A) ingredients in perfume compositions Aramis and Athos.

For comparison, similar tests were also performed on a typical household cleaning product perfume with the following composition:

Table 4 Typical household cleaning product perfume

- Ingredient	100% <sup>w</sup> / <sub>w</sub>			
ALDEHYDE C10 (DECANAL)	5			
CAMPHOR POWDER SYNTHETIC				
(1,7,7-trimethylbicyclo[2.2.1]heptan-2-	0.2			
one)				
CINEOLE PQ				
(1,3,3-trimethyl-2-	2.5			
oxabicyclo[2.2.2]octane)				
CIS 3 HEXENOL				
((3Z)-hex-3-en-1-ol)(10% solution in	0.3			
DPG)*				
CITRONELLOL PURE	5			
(3,7-dimethyloct-6-en-1-ol)	,			
DIHYDRO MYRCENOL (Q)	19			
(2,6-dimethylhept-5-en-2-ol)				
DIPROPYLENE GLYCOL	27.5			
(1-[(2-hydroxypropyl)oxy]propan-2-ol)	27.0			
ETHYL BUTYRATE	0.5			
(10% solution in DPG)(ethyl butanoate)				
GERANIOL 70	- 5			
((2E)-3,7-dimethylocta-2,6-dien-1-ol)				
HEXYL CINNAMIC ALDEHYDE	5			
((2E)-2-hexyl-3-phenylprop-2-enal)	J			

Percent detachment value (PDV)	-43
yl)ethyl acetate)	•
(1-methyl-1-(4-methylcyclohex-3-en-1-	2
TERPINYL ACETATE	
methylethylidene)cyclohex-1-ene)	
(1-methyl-4-(1-	2
TERPINOLENE EXTRA	
2-oI)	
(2-(4-methylcyclohex-3-en-1-yl)propan-	. 1
TERPINEOL	
(2-phenylethanol)	0.0
PHENYL ETHYL ALCOHOL	0.5
ORANGE BRAZIL PURE	2
ORANGE BOOSTER B 5535 (Q)	1.5
((2Z)-3,7-dimethylocta-2,6-dien-1-ol)	5
NEROL STD	3
pentylcyclopentyl]acetate)	
(methyl [(1R,2S)-3-oxo-2-	4.5
SUPER (Q)	4.5
METHYL DIHYDRO JASMONATE	
carbaldehyde)	
(2,4-dimethylcyclohex-3-ene-1-	1 .
LIGUSTRAL (Q)	
LEMONGRASS oil	1
octahydronaphthalen-2-yl)ethanone)	
(1-(2,3,8,8-tetramethyl-1,2,3,4,5,6,7,8-	0.5
ISO AMBOIS SUPER CI (Q)	<u> </u>
acetate)	
(1,7,7-trimethylbicyclo[2.2.1]hept-2-yl	11
ISO BORNYL ACETATE	

\*DPG = dipropylene glycol

Q indicates material available from Quest International.

#### Example 6

### Effect of increasing levels of antagonist perfume in active perfume compositions

The robustness of biofilm detaching performance of perfume compositions Aramis and Athos of Example 3 (with 25% of active perfume ingredients) in the presence of increasing amounts of antagonist perfume Porthos (5-25% of final perfume concentration) were investigated. The balance of the perfume formulation consisted of non-active (Group B) ingredients. The final perfume concentration was 0.25%, prepared in quarter strength GPC base. These data are presented in figure 2 and the perfume formulations are listed in Tables 5, 6 and 7.

Table 5 Perfume composition Aramis (25% of final perfume concentration):

Group	Ingredient	% by weight (total ingredients $100\%$ "/ <sub>w</sub> )			ents	
		0	5	10	15	25
В	Rose fragrance 0409™ (available from Quest International)	8.3	7.8	7.2	6.7	5.6
В	Ethyl-2-methyl-1,3-dioxolan-2-yl acetate	4.2	3.9	3.6	3.3	2.8
В	5-heptyldihydrofuran-2(3H)-one	2.1	1.9	1.8	1.7	1.4
В	Origanum	10.4	9.7	9.0	8.3	6.9
В	1-(methyloxy)propan-2-ol	10.4	9.7	9.0	8.3	6.9
В	2,2-dimethyl-3-(3- methylphenyl)propan-1-ol	6.3	5.8	5.4	5.0	4.2
В	tricyclo[5.2.1.0 <sup>4</sup> (2,6)]dec-4-en-8-yl propanoate	10.4	9.7	9.0	8.3	6.9
В	1,7,7- Trimethylbicyclo[2.2.1]hept-2- yl acetate	4.2	3.9	3.6	3.3	2.8
В	2-{[2- (Ethyloxy)ethyl]oxy}ethanol	10.4	9.7	9.0	8.3	6.9
В	3,7-Dimethyloct-6-en-1-ol	6.3	5.8	5.4	5.0	4.2
В	3a,6,6,9a-	2.1	1.9	1.8	1.7	1.4

	tetramethyldodecahydronaphtho [2,1-{b}]furan					. <del>-</del>
A	1-Methyl-4-(1-methylethyl)-2- [(1E)-prop-1-enyl]benzene	8.9	8.9	8.9	8.9	8.9
A	3-methylcyclopentadecanone	7.1	7.1	7.1	7.1	7.1
A	ethyl 3-(1- methylethyl)bicyclo[2.2.1]hept- 5-ene-2-carboxylate	5.4	5.4	5.4	5.4	5.4
A	Ylang Ylang oil	3.6	3.6	3.6	3.6	3.6
С	2-methyl-3-[4-(methyoxy) phenyl] propanal	0.0	1.8	3.6	5.4	8.9
C	(2E)-3-phenylprop-2-enal	0.0	1.4	2.9	4.3	7.1
C	lemongrass oil	0.0	1.1	2.1	3.2	5.4
C	1,3-dimethylbut-3-enyl 2-methylpropanoate	0.0	0.7	1.4	2.1	3.6
	PDV	34	28	18	15	-9

Table 6 Perfume composition Athos (25% of final perfume concentration):

Group	Ingredient	% by weight (total ingredients 100%"/w)			nts	
		0	5	10	15	25
В	Rose fragrance 0409™ (available from Quest International)	8.3	7.8	7.2	6.7	5.6
В	Ethyl-2-methyl-1,3-dioxolan-2-yl acetate	4.2	3.9	3.6	3.3	2.8
В	5-heptyldihydrofuran-2(3H)- one	2.1	1.9	1.8	1.7	1.4
В	Origanum	10.4	9.7	9.0	8.3	6.9
В	1-(methyloxy)propan-2-ol	10.4	9.7	9.0	8.3	6.9
В	2,2-dimethyl-3-(3-methylphenyl)propan-1-ol	6.3	5.8	5.4	5.0	4.2
В	tricyclo[5.2.1.0^{2,6}]dec-4-en-8-yl propanoate	10.4	9.7	9.0	8.3	6.9
В	1,7,7- Trimethylbicyclo[2.2.1]hept- 2-yl acetate	4.2	3.9	3.6	3.3	2.8
В	2-{[2- (Ethyloxy)ethyl]oxy}ethanol	10.4	9.7	9.0	8.3	6.9
В	3,7-Dimethyloct-6-en-1-ol	6.3	5.8	5.4	5.0	4.2
В	3a,6,6,9a- tetramethyldodecahydronapht ho[2,1-{b}]furan	2.1	1.9	1.8	1.7	1.4
A	(2E)-tridec-2-enenitrile	8.9	8.9	8.9	8.9	8.9

A	1-(2,6,6,8- tetramethyltricyclo[5.3.1.0 <sup>4</sup> 1,5}]undec-8-en-9- yl)ethanone	7.1	7.1	7.1	7.1	7.1
A	1-methylethyl tetradecanoate	5.4	5.4	5.4	5.4	5.4
A	(2Z)-2-ethyl-4-(2,2,3- trimethylcyclopent-3-en-1- yl)but-2-en-1-ol	3.6	3.6	3.6	3.6	3.6
С	2-methyl-3-[4-(methyoxy) phenyl] propanal	0.0	1.8	3.6	5.4	8.9
С	(2E)-3-phenylprop-2-enal	0.0	1.4	2.9	4.3	7.1
C	lemongrass oil	0.0	1.1	2.1	3.2	5.4
С	1,3-dimethylbut-3-enyl 2-methylpropanoate	0.0	0.7	1.4	2.1	3.6
	PDV	54	37	33	25	2

Table 7 Non-active perfume

Group	Ingredient	% by weight (total ingredients $100\%$ ,				its
		0	5	10	15	25
В	Rose fragrance 0409 <sup>TM</sup> (available from Quest International)	11.1	10.6	10.0	9.4	8.3
В	Ethyl-2-methyl-1,3-dioxolan-2-yl acetate	5.6	5.3	5.0	4.7	4.2
В	5-heptyldihydrofuran-2(3H)- one	2.8	2.6	2.5	2.4	2.1
В	Origanum	13.9	13.2	12.5	11.8	10.4
В	1-(methyloxy)propan-2-ol	13.9	13.2	12.5	11.8	10.4
В	2,2-dimethyl-3-(3- methylphenyl)propan-1-ol	8.3	7.9	7.5	7.1	6.3
· В	tricyclo[5.2.1.0^{2,6}]dec-4-en-8-yl propanoate	13.9	13.2	12.5	11.8	10.4
В	1,7,7- Trimethylbicyclo[2.2.1]hept- 2-yl acetate	5.6	5.3	5.0	4.7	4.2
В	2-{[2- (Ethyloxy)ethyl]oxy}ethanol	13.9	13.2	12.5	11.8	10.4
В	3,7-Dimethyloct-6-en-1-ol	8.3	7.9	7.5	7.1	6.3
В	3a,6,6,9a- tetramethyldodecahydronapht ho[2,1-{b}]furan	2.8	2.6	2.5	2.4	2.1

	PDV	28	18	-27	-14	-29
	2-methylpropanoate	0.0	0.7	1.4	2.1	3.0
C	1,3-dimethylbut-3-enyl	0.0	0.7	1.4	2.1	3.6
С	lemongrass oil	0.0	1.1	2.1	3.2	5.4
С	(2E)-3-phenylprop-2-enal	0.0	1.4	2.9	4.3	7.1
С	2-methyl-3-[4-(methyoxy) phenyl] propanal	0.0	1.8	3.6	5.4	8.9

These results are summarised in Figure 2.

### Example 7

## Perfumes prepared in de-ionised water

15 component perfume formulations (including 11 non-active (Group B) ingredients) containing either four active (Group A) or 4 antagonist (Group C) ingredients (at 50% of final perfume concentration) were prepared in de-ionised water. The final perfume concentration was  $0.25\,\%^{\rm w}/_{\rm w}$ . The perfume formulations are listed (Table 8) together with percent detachment values. Results are shown in Figure 3.

Table 8 Perfume formulations prepared in de-ionised water

Group	Ingredients		ne formı 100%"/,	
		Aramis	Athos	Porthos
В	Rose fragrance 0409™ (available from Quest International)	5.6	5.6	5.6
В	Ethyl-2-methyl-1,3-dioxolan-2-yl acetate	2.8	2.8	2.8
В	5-heptyldihydrofuran-2(3H)-one	1.4	1.4	1.4 .
В	Origanum	6.9	6.9	6.9
В	1-(methyloxy)propan-2-ol	6.9	6.9	6.9
В -	2,2-dimethyl-3-(3-methylphenyl)propan-1-ol	4.2	4.2	4.2
В	tricyclo[5.2.1.0 <sup>4</sup> [2,6]]dec-4-en-8-yl propanoate	6.9	6.9	6.9
В	1,7,7-Trimethylbicyclo[2.2.1]hept-2-yl acetate	2.8	2.8	2.8
В	2-{[2-(Ethyloxy)ethyl]oxy}ethanol	6.9	6.9	6.9
В	3,7-Dimethyloct-6-en-1-ol	4.2	4.2	4.2
В	3a,6,6,9a-	1.4	1.4	1.4

		<del></del>		
	tetramethyldodecahydronaphtho[2,1-			
	{b}]furan			
A	1-Methyl-4-(1-methylethyl)-2-[(1E)-prop-	17.9	0	0
	1-enyl]benzene	17.9	U	U
A	3-methylcyclopentadecanone	14.3	0	0
A	Ethyl 3-(1methylethyl)bicyclo[2.2.1]	10.7	0	0
	hept-5-ene-2-carboxylate	10.7	0	U
A	Ylang Ylang oil	7.1	0	0
A	(2E)-tridec-2-enenitrile	0	17.9	0
A	1-(2,6,6,8-			
	tetramethyltricyclo[5.3.1.0 <sup>1</sup> ,5]undec-	0	14.3	0
	8-en-9-yl)ethanone			
A	1-methylethyl tetradecanoate	0	10.7	0
A	(2Z)-2-ethyl-4-(2,2,3-trimethylcyclopent-	0	7.1	0
	3-en-1-yl)but-2-en-1-ol	U	/.1	U
С	2-methyl-3-[4-(methyoxy) phenyl]	0	0	17.0
	propanal	U	U	17.9
C	(2E)-3-phenylprop-2-enal	0	0	14.3
C	Lemongrass oil	0	0	10.7
C	1,3-dimethylbut-3-enyl 2-	0	0	7.1
	methylpropanoate	0	U	7.1
	Percent detachment value (PDV)	40	35	-10

These results are shown in Figure 3.

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#### **CLAIMS**

- 1. A perfume composition comprising at least 15% by weight of at least one Group A perfume ingredient, and wherein Group A and Group B ingredients together constitute at least 80% by weight of the perfume composition.
- 2. A perfume composition according to claim 1, wherein the Group A ingredients are selected from the group consisting of (2E)-tridec-2-enenitrile, (2Z)-2-ethyl-4-(2,2,3trimethylcyclopent-3-en-1-yl)but-2-en-1-ol, (3E)-3-methyl-4-(2,6,6-trimethylcyclohex-2-en-1-yl)but-3-en-2-one, 1-(2,3,8,8-tetramethyl-1,2,3,5,6,7,8,8a-octahydronaphthalen-2yl)ethanone, 1-(2,6,6,8-tetramethyl-tricyclo[5.3.1.0^{1.5}]undec-8-en-9-vl)ethanone, 1-(methyloxy)-4-[(1E)-prop-1-enyl]benzene, 1,4-dioxacycloheptadecane-5.17-dione, 1-[1,1,2,6-tetramethyl-3-(1-methylethyl)-2,3-dihydro-1H-inden-5-yl]ethanone, 1-methyl-4-(1methylethyl)-2-[(1E)-prop-1-enyl]benzene, 1-methylethyl tetradecanoate, 2-(2methylpropyl)-4-hydroxy-4-methyl-tetrahydropyran, 2,6,10-trimethylundec-9-enal, 2-[2-(4methylcyclohex-3-en-1-yl)propyl]cyclopentanone, 2-methylundecanal, 5-methyl-2-(1methylethyl)phenol, anisic aldehyde, cinnamic alcohol, clove bud rectified extra DQ P353, cyclohexadecanolide, cyclopentadecanone, decanol DQ, dodecanenitrile, eucalyptol, eucalyptus globulus, eugenol rectified, ginger oils, isoamyl acetate, nonanol, patchouli oil, phenylmethanol, propane-1,2-diol, tea tree oil DQ, tributyl-2-(acetyloxy)propane-1,2,3tricarboxylate, 1H-indole, (2E)-2-pentyl-3-phenylprop-2-enal, (4E)-4-tricyclo[5.2.1.0<sup>2</sup>,6] dec-8-ylidenebutanal, 2,6,6,8tetramethyltricyclo[5.3.1.0^{1,5}]undec-8-yl acetate, 2-[(2-{[2-(methyloxy)propyl]oxy}propyl)oxy]propan-1-ol, 2-methyldecanenitrile, 1-(1,1,2,3,3,6-hexamethyl-2,3-dihydro-1H-inden-5-yl)ethanone, 1-(3,5,5,6,8,8-hexamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)ethanone, 4-(1,7,7-trimethylbicyclo[2.2.1]hept-2-yl)cyclohexanol, Silvanone (Silvanone is a Trade Mark), 2-heptylcyclopentanone, 3-methyldodecanenitrile, (6E)-3,7,11-trimethyldodeca-1,6,10-trien-3-ol, methyl (2E)-non-2-enoate, (2E)-2-hexyl-3-phenylprop-2-enal, ethyl 3-(1-methylethyl)bicyclo[2.2.1]hept-5-ene-2-carboxylate, 3methylcyclopentadecanone, ylang ylang oil, 2-(1,1-dimethylethyl)cyclohexyl acetate,

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2,4-dimethyl-4,4a,5,9b-tetrahydroindeno[1,2-{d}][1,3]dioxine, 4,6,6,7,8,8-hexamethyl-1,3,4,6,7,8-hexahydroindeno[5,6-{c}]pyran (e.g. in the form of Galaxolide – Galaxolide is a Trade Mark), [4-(1-methylethyl)cyclohexyl]methanol, capsicum oleoresin DQ, peppermint American lagrande DQ, lime terpeneless DQ, peppermint American yakima rectified DQ, citronella Ceylon DQ, peppermint arvensis co-distilled DQ, rose flavour base ABF0339A, marjoram French DQ, jasmin absolute DQ, coriander DQ, cinnamic aldehyde extra DQ, citral, lavender oil DQ, iso propyl alcohol DQ, amyl cinnamic aldehyde DQ amyl caproate DQ, peppermint Chinese triple rectified (DQ), and ketones of general formula RCOR' having an octanol-water partition coefficient of at least 4 (expressed as a logarithm to base 10), where R and R' are independently hydrocarbyl residues that may be aliphatic or aromatic, saturated or unsaturated, and combinations thereof, but may not contain other functional groups, including 1-(2,3,8,8-tetramethyl-1,2,3,4,5,6,7,8-octahydronaphthalen-2yl)ethanone, 1-(2,6,6,8-tetramethyltricyclo[5.3.1.0^{1,5}]undec-8-en-9-yl)ethanone, 1-(1,1,2,3,3,6-hexamethyl-2,3-dihydro-1H-inden-5-yl)ethanone, 1-(3,5,5,6,8,8-hexamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)ethanone, 3-methylcyclopentadecanone, 1-[1,1,2,6tetramethyl-3-(1-methylethyl)-2,3-dihydro-1H-inden-5-yl]ethanone

3. A perfume composition according to claim 1 or claim 2, wherein the Group B ingredients are selected from the group consisting of (3Z)-hex-3-en-1-o, 1,7,7-trimethylbicyclo[2.2.1]hept-2-yl acetate, 1-methyl-4-(1-methylethylidene)cyclohex-1-ene, 2-(phenyloxy)ethanol, 2,6,10-trimethyl-1-acetyl-cyclododeca-2,5,9-triene, 2-{[2-(ethyloxy)ethyl]oxy}ethanol, 3,7-dimethyloct-6-en-1-ol, diethyl benzene-1,2-dicarboxylate, (4E)-dec-4-enal, ethyl-2-methyl-1,3-dioxolan-2-yl acetate, 5-heptyldihydrofuran-2(3H)-one, origanum, 1-(methyloxy)propan-2-ol, 2,2-dimethyl-3-(3-methylphenyl)propan-1-ol, tricyclo[5.2.1.0^{2,6}]dec-4-en-8-yl propanoate, 3a,6,6,9a-tetramethyldodecahydronaphtho[2,1-{b}]furan, hexyl 2-hydroxybenzoate, phenylmethyl-2-hydroxybenzoate, cyclohexadec-5-en-1-one, Rose fragrance 0409™ (available from Quest International), 1-ethenyl-1,5-dimethylhex-4-enyl acetate, (4Z)-dec-4-enal, 1-(5,5-dimethylcyclohex-1-en-1-yl)pent-4-en-1-one, 3-methyl-1-(2-methylpropyl)butyl acetate, 2-(methyloxy)-4-propylphenol, 2-(acetyloxy)-1-[(acetyloxy)methyl]ethyl acetate, Thyme Red, 4-(1,1-dimethylethyl)cyclohexyl acetate, 5-methyl-2-(1-methylethyl)cyclohexanol, Aldehyde C10 DQ, Lime Terpenes Washed DQ, Peppermint Piperita Indian Rectified FDC DQ, Anisic

aldehyde, Blackcurrent Base ABF0972, Peppermint Indian Rectified (FDC) DQ, Grapefruit DQ, Parsley Herb, Peppermint American Far West Rectified DQ, Orange Terpenes Ex Concentrate DQ, Iso Amyl Butyrate DQ, Cinnamon Supra ABF1092, Cardamom English Distilled DQ, Apple Base ABF1016, Peppermint Chinese Terpeneless DQ, Peppermint Moroccan DQ, Peppermint American Idaho DQ, Orange Terpeneless DQ,

- 4. A perfume composition according to any one preceding claim, comprising at least 3 or at least 3 Group A perfume ingredients.
- 5. A perfume composition according to any one preceding claim, comprising at least 25% by weight, preferably at least 30% by weight, preferably at least 40% by weight or possibly at least 60% by weight of Group A perfume ingredients.
- A perfume composition according to any one of the preceding claims, further comprising at least one Group B perfume ingredient.
- 7. A perfume composition according to any one preceding claim, comprising at least 3 Group B perfume ingredients.
- 8. A perfume composition according to any one preceding claim, which comprises at most 15 % by weight of Group C ingredients.
- 9. A perfume composition according to claim 8, wherein the Group C ingredients are selected from the group consisting of 2-methyl-3-[4-(methyoxy) phenyl] propanal, (2E)-3phenylprop-2-enal, lemongrass oil, 1,3-dimethylbut-3-enyl 2-methylpropanoate, Muguet base AB7001<sup>TM</sup>, Moss base AB7004<sup>TM</sup>, Sandalone AC802<sup>TM</sup>, Jasmin AB7002<sup>TM</sup>, prop-2-enyl [(2-methylbutyl)oxy]acetate, 2,6-dimethylhept-5-en-2-ol, phenylmethyl acetate, Carvone, 3pentyltetrahydro-2H-pyran-4-yl acetate, 3,7-dimethylocta-1,6-dien-3-ol, 4-(4-hydroxy-4methylpentyl)cyclohex-3-ene-1-carbaldehyde, methyl (3-oxo-2-pentylcyclopentyl)acetate, 3-methyl-5-phenylpentan-1-ol, (1E)-1-(2,6,6-trimethylcyclohex-2-en-1-yl)pent-1-en-3-one, 2-phenylethanol, 2-(4-methylcyclohex-3-en-1-yl)propan-2-ol, Orange oil. 1-[(2hydroxypropyl)oxy]propan-2-ol, (6Z)-3,7-dimethylnona-1,6-dien-3-ol.

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- 10. A perfume composition according to any one of the preceding claims, wherein each perfume ingredient is present in an amount not exceeding 20% by weight.
- 11. A perfume composition according to any one of the preceding claims, wherein at least one of the perfume ingredients has antimicrobial properties.
- 12. A perfumed product comprising a perfume composition in accordance with any one of the preceding claims.
- 13. A perfumed product according to claim 12, which is a household cleaning product.
- 14. A perfumed product according to claim 12, which is a dental care product.
- 15. A perfumed product according to claim 12, which is a deodorant product.
- 16. A method of detaching a biofilm from a surface, comprising application to the surface of a perfume composition or perfumed product in accordance with any one of claims 1 to 12.
- 17. A method according to claim 16, wherein the surface is a household surface.
- 18. A method according to claim 16, wherein the surface is an oral surface.
- 19. A method according to claim 16, wherein the surface is a human skin surface.
- 20. Use of a perfume ingredient in Group A for the purpose of detaching a biofilm.

Figure 1

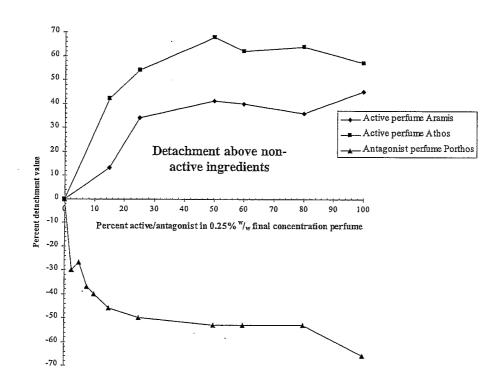


Figure 2

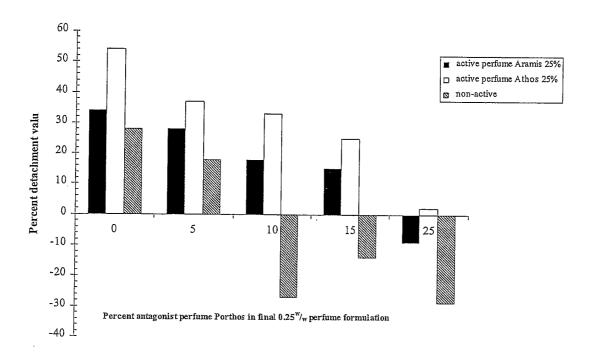
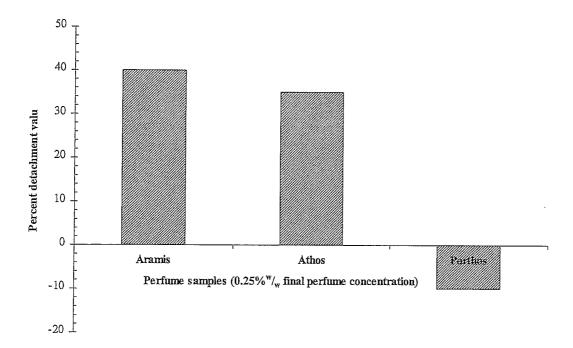


Figure 3



### PATENT COOPERATION TREATY

# **PCT**

### INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	FOR FURTHER	see Form PCT/ISA/220		
HCM/319.01/Q	ACTION as	well as, where applicable, item 5 below.		
International application No.	International filing date (day/month/year)	(Earliest) Priority Date (day/month/year)		
PGE /GP 2 2 0 C / 2 2 1 2 C F	13/04/2006 14/04/2005			
PCT/GB2006/001365 Applicant	13/04/2006	14/04/2005		
Applicant				
OTTECH THEEDMANTONAL CEDIT	710 D 17			
QUEST INTERNATIONAL SERVICE	LES D.V.			
This international search report has been according to Article 18. A copy is being tra		uthority and is transmitted to the applicant		
This international search report consists of	of a total of 6 sheets.			
It is also accompanied by	a copy of each prior art document cited in	this report.		
Basis of the report				
a. With regard to the language, the	international search was carried out on the	basis of:		
=	application in the language in which it was			
a translation of th of a translation fu	e international application into rnished for the purposes of international se	, which is the language arch (Rules 12.3(a) and 23.1(b))		
b. X With regard to any nucle	otide and/or amino acid sequence disclo	sed in the international application, see Box No. I.		
2. X Certain claims were fou	nd unsearchable (See Box No. II)			
3. Unity of invention is lac	king (see Box No III)			
4. With regard to the title,		• • •		
X the text is approved as su	bmitted by the applicant			
the text has been establis	hed by this Authority to read as follows:			
,				
	•	•		
5. With regard to the abstract,				
X the text is approved as su	bmitted by the applicant			
		thority as it appears in Box No. IV. The applicant search report, submit comments to this Authority		
,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	-			
6. With regard to the drawings,				
a. the figure of the drawings to be a	oublished with the abstract is Figure No3	-/1		
. X as suggested by	the applicant			
as selected by th	is Authority, because the applicant failed to	suggest a figure		
as selected by th	is Authority, because this figure better chai	acterizes the invention		
b. none of the figures is to b	e published with the abstract			

## INTERNATIONAL SEARCH REPORT

International application No PCT/GB2006/001365

			1/ 602000/001365		
A. CLASS INV.	A. CLASSIFICATION OF SUBJECT MATTER INV. C11B9/00				
According t	o International Patent Classification (IPC) or to both national classifi	cation and IPC			
<del></del>	SEARCHED				
	ocumentation searched (classification system followed by classification sy	tion symbols)			
Documenta	tion searched other than minimum documentation to the extent that	such documents are included	in the fields searched		
Electronic c	data base consulted during the international search (name of data b	ase and, where practical, sear	ch terms used)		
EPO-In	ternal				
C. DOCUM	ENTS CONSIDERED TO BE RELEVANT				
Category*	Citation of document, with indication, where appropriate, of the re	elevant passages	Relevant to claim No.		
X	WO 00/57699 A (INNOSCENT LTD; RO NEVO, MELVYN) 5 October 2000 (20	SENBERG 00-10-05)	1-6,11, 12,14, 16,18		
А	example 1		7-10,13, 15,17, 19,20		
A	WO 2004/073670 A (QUEST INTERNATIONAL SERVICES B.V; BEHAN, JOHN, MARTIN; BRADSHAW, DAVID) 2 September 2004 (2004-09-02) page 8, paragraph 4; claim 10; examples 6,8		1-20		
<b>A</b>	EP 1 238 650 A (TAKASAGO INTERNATIONAL CORPORATION) 11 September 2002 (2002-09-11) claims 1-5; example 2		1-20		
		<b>-</b> /			
X Furti	her documents are listed in the continuation of Box C.	X See patent family ar	inex.		
*Special categories of cited documents:  *A* document defining the general state of the art which is not considered to be of particular relevance  *E* earlier document but published on or after the International filing date  *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)  *O* document referring to an oral disclosure, use, exhibition or other means  *P* document published after the international filing date or priority date and not in conflict with the application but clied to understand the principle or theory underlying the invention cannot be considered novel or cannot be considered to involve an inventive step when the document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined with one or more other such document is combined invention.		n conflict with the application but principle or theory underlying the levance; the claimed invention over the considered to owhen the document is taken alone levance; the claimed invention involve an inventive step when the with one or more other such docunning obvious to a person skilled			
Date of the actual completion of the international search  Date of mailing of the international search report			ernational search report		
26 July 2006 03/08/2006					
Name and n	mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2	Authorized officer			
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016  Saunders, T			τ .		

#### INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2006/001365

C(Continus	tion). DOCUMENTS CONSIDERED TO BE RELEVANT	PCT/GB2006/001365
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 6 585 961 B1 (STOCKEL RICHARD F) 1 July 2003 (2003-07-01) column 1, line 4 - line 14; claim 1	1-20
Α	MORRIS J A ET AL: "ANTIMICROBIAL ACTIVITY OF AROMA CHEMICALS AND ESSENTIAL OILS" JOURNAL OF THE AMERICAN OIL CHEMISTS' SOCIETY, AOCS PRESS, CHAMPAIGN, IL, US, 1 May 1979 (1979-05-01), pages 595-603,	1–20
	XP000645444 ISSN: 0003-021X cited in the application table III	
		•

#### FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box II.2

The present claim 1 relates to an extremely large number of possible compounds. Support and disclosure in the sense of Article 6 and 5 PCT is to be found however for only a very small proportion of the compounds claimed (cf. pages 4-12). The non-compliance with the substantive provisions is to such an extent, that the search was performed taking into consideration the non-compliance in determining the extent of the search of claim 1 (PCT Guidelines 9.19 and 9.23).

The search of claim 1 was restricted to the general concept of the use of perfume compositions for removing biofilms from surfaces, as reflected on page 4.

It is noted that a perfume is defined as belonging to a Group A or Group B on the basis of its biofilm removal capability, expressed in terms of percent detachment values (PDV's).

The use of this unusual parameter (PDV) in the present context is considered to lead to a lack of clarity because the claim does not clearly identify the products encompassed by it as the parameters cannot be clearly and reliably determined by indications in the description or by objective procedures which are usual in the art. This makes it impossible to compare the claims to the prior art. As a result, the application does not comply with the requirement of clarity under Article 6 PCT.

The same reasoning can be applied to independent claims 12, 16 and 20.

The applicant's attention is drawn to the fact that claims relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure. If the application proceeds into the regional phase before the EPO, the applicant is reminded that a search may be carried out during examination before the EPO (see EPO Guideline C-VI, 8.5), should the problems which led to the Article 17(2) declaration be overcome.

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### INTERNATIONAL SEARCH REPORT

Box II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)
This international Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:
Claims Nos.:     because they relate to subject matter not required to be searched by this Authority, namely:
2. X Claims Nos.:  because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:  see FURTHER INFORMATION sheet PCT/ISA/210
3. Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).
Box III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)
This International Searching Authority found multiple inventions in this international application, as follows:
As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
4. No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:
Remark on Protest  The additional search fees were accompanied by the applicant's protest.  No protest accompanied the payment of additional search fees.

#### INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No PCT/GB2006/001365

Patent document cited in search report		Publication date	Patent family member(s)		Publication date	
WO 0057699	Α	05-10-2000	AU EP JP US	3322000 1162883 2002540123 2002064505	A1 T	16-10-2000 19-12-2001 26-11-2002 30-05-2002
WO 2004073670	Α	02-09-2004	EP US	1594452 2006153958		16-11-2005 13-07-2006
EP 1238650	Α	11-09-2002	JP US	2002265978 2003039616		18-09-2002 27-02-2003
US 6585961	B1	01-07-2003	NONE			