

EUROPEAN QUALIFYING EXAMINATION 2007

PAPER B CHEMISTRY

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Annex 1 (Patent Application)

Carpet Shampoo

5 [0001] The present invention relates to a carpet shampoo. A carpet shampoo is a dilute aqueous solution used to clean carpets. Carpet shampoos are mainly used for permanently installed carpets and are usually applied and worked into the carpet using a special device with rotating brushes. The shampoo is then allowed to dry to a non-tacky solid residue which can be removed by vacuum cleaning.

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[0002] Modern carpets are overwhelmingly produced from polyamide and polyester fibres. A carpet made from these fibres is hard-wearing and resistant to soiling and staining. New carpets are frequently treated with a fluorocarbon polymer to provide a further improvement in stain resistance. All carpets however become soiled and stained
15 with time. The soil in the carpet contains dust and dirt particles which have become firmly attached to the carpet fibres. Carpets are frequently stained by coffee, wine, food, grease or oils.

[0003] Carpet shampoos are very effective at removing soil and stains from a carpet. A
20 significant problem with carpet shampooing is that the cleaning process often degrades the soil and stain resistance of the carpet. Thus the more a carpet is cleaned the quicker it becomes dirty and once again requires cleaning.

[0004] Carpets have in recent years also been recognised as a significant problem for
25 people allergic to dust mites. The number of dust mites in the carpet of a modern centrally heated house may be very large and this results in a very large concentration of allergens in the house. It would be desirable to provide a carpet shampoo able to reduce the number of dust mites in the carpet.

[0005] It is also important that the carpet shampoo contains no toxic ingredients and dries to a residue which is easily removed from the carpet.

[0006] The present invention provides a carpet shampoo able to address these
5 problems. The composition consists of:

5 - 20 wt.% of surfactant

1 - 8 wt.% of a resoiling inhibitor

0.1 - 5 wt.% of an acaricide

10 1 - 2 wt.% of a sequestering agent

0 - 5 wt.% of an organic solvent

and balance water

[0007] The surfactants used in the shampoo may be anionic, cationic or non-ionic. Best
15 results are however obtained when at least 50 wt.% of the surfactants are anionic. The surfactants used are typically mixtures of at least one anionic and at least one non-ionic surfactant. Examples of suitable anionic surfactants include ammonium salts of alkyl sulphonates, sodium alkyl sulphates and disodium lauryl sulphosuccinate. A particularly preferred class of anionic surfactants are sodium alkylsulphates with 12 to 16 carbon
20 atoms. Preferred non-ionic surfactants are ethoxylated long chain alcohols and ethoxylated nonylphenols.

[0008] Resoiling inhibitors are polymers which, when used in carpet shampoos, minimise the degradation of the stain and soil repellent properties of the carpet. The
25 resoiling inhibitors are preferably selected from polyacrylates, polyacrylic acid and styrene-maleic acid copolymers. A particularly preferred resoiling inhibitor is a fluorinated polyacrylate containing monomer units of formula (I):



This preferred resoiling inhibitor is commercially available as NoDirt.

5 [0009] The carpet shampoo of the invention also contains an acaricide. Acaricides kill dust mites. Acaricides useful in the present invention are required to be non-toxic. The acaricide used in the present invention may be selected from benzyl benzoate, phenyl salicylate and essential oils. An essential oil is a water-immiscible liquid obtained by distilling plant material. The only essential oils which are acaricides are deerhorn cedar oil, lemon oil, rosemary oil and thyme oil. These essential oils only have an acaricidal effect in shampoos in which at least 50 wt.% of the surfactants are anionic surfactants. Essential oils are less effective acaricides than benzyl benzoate and phenyl salicylate. The essential oils are however less toxic than benzyl benzoate and phenyl salicylate.

15 [0010] A carpet shampoo formulated with hard water is much less effective at removing soil from a carpet than a shampoo formulated with deionised water. In order to counteract the effect of water hardness, a sequestering agent is used. The most commonly used sequestering agent is the sodium salt of ethylenediamine tetraacetic acid (Na-EDTA). Nitrilotriacetic acid salts may also be used.

20 [0011] An organic solvent is present in the carpet shampoo if phenyl salicylate or benzyl benzoate is used as the acaricide. These materials are solids at room temperature and it is not possible to dissolve a sufficient concentration of these compounds in the shampoo without an organic solvent. The organic solvent may be a glycol ether. No organic solvent is used if the acaricide is an essential oil. Carpet shampoos containing no organic solvents are preferred for environmental reasons.

[0012] If lemon oil and deerhorn cedar oil are used as acaricides then the composition will have a fragrance. All of the components necessary to make the carpet shampoo of the invention are commercially available.

- 5 [0013] The shampoo, once applied to the carpet, must be allowed to dry. Typically a carpet which has been shampooed using a modern carpet-cleaning device will dry in about 4 hours. The dry residue can then be removed by vacuum cleaning. If the acaricide is an essential oil it is however necessary to leave the residue for at least 12 hours after the carpet has been shampooed. This extra time is indispensable if the
10 essential oil is to kill a significant number of dust mites.

Examples:

[0014] Example 1: A carpet shampoo was prepared containing the following ingredients:

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10 wt.% of XQsurf

2 wt.% of Detnon

2 wt.% of NoDirt

1 wt.% of benzyl benzoate

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1.2 wt.% of Na-EDTA

1 wt.% of 2-hexyloxyethanol (a glycol ether solvent)

and balance water

- 25 XQsurf is a commercially available anionic surfactant mainly containing sodium alkyl sulphates with 12 - 16 carbon atoms. Detnon is a non-ionic surfactant containing ethoxylated nonylphenols. NoDirt is a commercially available resoiling inhibitor.

[0015] Example 2: A carpet shampoo was prepared containing the following ingredients:

5 wt.% of XQsurf

5 wt.% of Detnon

5 2 wt.% of NoDirt

1 wt.% of phenyl salicylate

1.2 wt.% of Na-EDTA

2 wt.% of 2-(2-ethoxyethoxy)ethanol (a glycol ether solvent)

and balance water

10

[0016] Example 3: A carpet shampoo was prepared containing the following ingredients:

8 wt.% of XQsurf

5 wt.% of Detnon

15 2 wt.% of NoDirt

1 wt.% of lemon oil

1.2 wt.% of Na-EDTA

and balance water

20 [0017] Example 4: A carpet shampoo was prepared containing the following ingredients:

10 wt.% of XQsurf

1 wt.% of Detnon

2 wt.% of NoDirt

25 0.5 wt.% of thyme oil

1.2 wt.% of Na-EDTA

and balance water

[0018] Example 5:

The carpet shampoos of examples 1 to 4 were each employed in a rotating brush type carpet-cleaning machine (PRACET 2000). The composition was left on the carpet for 14 hours and the carpet was then vacuum cleaned. The carpet shampoos provided an excellent cleaning performance and reduced the number of dust mites in the carpets by over 50%. The treated carpets also exhibited an excellent resistance to soiling and staining.

Claims:

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[0019] 1. A carpet shampoo consisting of:

5-20 wt.% of surfactant

1-8 wt.% of a resoiling inhibitor

15 0.1-5 wt.% of an acaricide

1-2 wt.% of a sequestering agent

0-5 wt.% of an organic solvent

and balance water

20 [0020] 2. The carpet shampoo of claim 1 where the surfactant is a mixture of at least one anionic and at least one non-ionic surfactant.

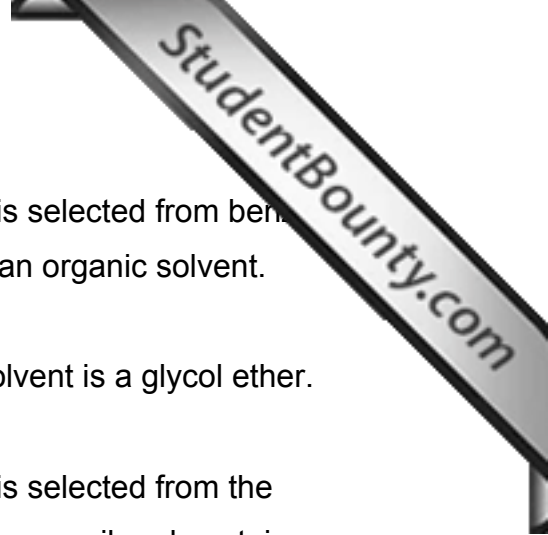
[0021] 3. The carpet shampoo of claim 1 where the surfactant mixture contains at least 50 wt.% of anionic surfactants.

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[0022] 4. The carpet shampoo of claim 1 where the resoiling inhibitor is a fluorinated polyacrylate, containing monomer units of formula (I):



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[0023] 5. The carpet shampoo of claim 1 where the acaricide is selected from benzyl benzoate and phenyl salicylate and the composition contains an organic solvent.

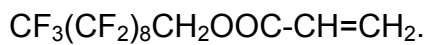
[0024] 6. The carpet shampoo of claim 5 where the organic solvent is a glycol ether.

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[0025] 7. The carpet shampoo of claim 3 where the acaricide is selected from the essential oils thyme oil, deerhorn cedar oil, lemon oil and rosemary oil and contains no organic solvent.

Annex 2 (Communication)

Document 1 (see example, and paragraphs [0002] to [0007]) discloses a carpet shampoo which contains 11 wt.% of surfactants (consisting of 10 wt.% of an anionic surfactant and 1 wt.% of a non-ionic surfactant), 2 wt.% of a resoiling inhibitor (NoDirt) and 1.2 wt.% of a sequestering agent. The composition also contains 0.5 wt.% of lemon oil which, as is clear from the present application, is an acaricide. The composition does not contain an organic solvent. The resoiling inhibitor used is a polyacrylate containing monomer units of the formula



Document 1 is thus novelty destroying for the subject-matter of claims 1 - 4 and 7 (Articles 52(1), 54(1) and 54(2) EPC).

The closest prior art for the subject-matter of claims 5 and 6 is document 2 (see paragraphs [0002] to [0006]). This document discloses carpet shampoos containing 1 wt.% of benzyl benzoate or phenyl salicylate as an acaricide where the compound is added with a glycol ether solvent at a concentration of 2 wt.% of the shampoo. The subject-matter of claims 5 and 6 is novel with respect to document 2 since it does not disclose the precise composition of the carpet shampoo and in particular does not disclose a shampoo with the composition required by claim 1 of the application. The problem addressed by the application in view of document 2 is to provide suitable carpet shampoos for mixing with the acaricide. Document 2 does however indicate that the acaricide can be mixed with commercially available carpet shampoos and identifies that of document 1 as a typical carpet shampoo. It would thus be obvious to mix the acaricide of document 2 with a shampoo as disclosed in document 1 and thus to arrive at a carpet shampoo as defined in claims 5 and 6 of the application when seeking to solve the problem. The subject-matter of claims 5 and 6 therefore does not involve an inventive step (Articles 52(1) and 56 EPC)

If the applicant wishes to maintain the application, new claims should be filed which take into account the above objections. Care should be taken to ensure that the new claims comply with the requirements of the EPC in respect of novelty, inventive step, clarity and if necessary unity (Articles 54, 56, 84 and 82 EPC). The applicant should also ensure that any amendments do not introduce subject-matter which extends beyond the content of the application as originally filed (Article 123(2) EPC).

In the letter of reply, the difference between the new claims and the state of the art disclosed in documents 1 and 2 as well as its significance should be identified. In addition the invention should be presented in such a way that the technical problem being solved in view of the closest state of the art, the solution proposed to this problem, as well as the position of the applicant in respect of inventive step (Rule 27(1)(c) EPC and Guidelines C-IV, 9.5) can be clearly understood.

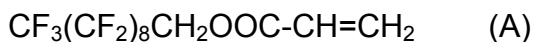
An independent claim must specify all the technical features necessary to define the invention (Guidelines C-III, 4.4). Thus each independent claim must contain all the technical features essential to the solution of the problem on which the invention is based.

In order to facilitate the examination as to whether the new claims contain subject-matter which extends beyond the content of the application as filed, the applicant is requested to indicate precisely where in the application documents any amendments proposed find a basis (Article 123(2) EPC, Guidelines E-II, 1 and C-VI, 5.4).

Annex 3 (Document 1)

Carpet cleaning composition with carpet protective properties

- 5 [0001] The present invention relates to a carpet cleaning composition. The carpet cleaning composition is particularly useful in carpet cleaning machines with rotating brushes. Carpet cleaning compositions of this type are commonly called carpet shampoos.
- 10 [0002] Carpet cleaning compositions which consist of an aqueous solution containing a surfactant, a sequestering agent, a fragrance and optionally an organic solvent are known. These compositions are very effective at cleaning carpets. However these cleaning compositions tend to degrade the anti-soiling and anti-stain properties of the carpet with the result that a carpet that has been cleaned with the known carpet cleaning
- 15 composition very quickly becomes dirty again. It has been proposed to address this problem by adding polyacrylic acid or a styrene-maleic acid copolymer to the composition. This is only partially successful and further improvements are still needed. It has been discovered that the degradation of the anti-soiling and anti-stain properties of the carpet during cleaning can be almost completely prevented by adding a polyacrylate
- 20 containing monomers of the formula A:



- [0003] The monomers of formula A must constitute at least 20 wt.% of the monomers in
- 25 the polyacrylate for it to be effective. The most preferred polymers contain 50 wt.% of monomers of formula A and 50 wt.% acrylic acid monomers. This polymer is commercially available as NoDirt. The polyacrylate is used at a concentration of 1 - 8 wt.%, preferably 2-3 wt.% of the carpet-cleaning composition.

[0004] The remainder of the carpet cleaning composition is conventional. The carpet cleaning composition contains surfactants, a sequestering agent, a fragrance and optionally an organic solvent.

5 [0005] The surfactants used in the shampoo may be anionic, cationic or non-ionic. Best results are however obtained when at least 50 wt.% of the surfactants are anionic. The surfactants used are mixtures of at least one anionic and at least one non-ionic surfactant. Examples of suitable anionic surfactants include ammonium salts of alkyl sulphates, sodium alkyl sulphates and disodium lauryl sulphosuccinate. A particularly
10 preferred class of anionic surfactants are sodium alkylsulphates with 12 to 16 carbon atoms. Preferred non-ionic surfactants are ethoxylated long chain alcohols and ethoxylated nonylphenols. The surfactants must be used at a concentration of at least 5 wt.% of the carpet cleaning composition. The maximum concentration of surfactants is typically 18 wt.%. Higher concentrations will still provide a good cleaning effect but are
15 uneconomical.

[0006] The sequestering agent is typically the sodium salt of ethylenediamine tetraacetic acid (Na-EDTA). Nitrilotriacetic acid salts may also be used. The sequestering agent is commonly used at a concentration of 1 - 3 wt.%. The organic solvent, if used, is typically
20 a glycol ether such as 2-hexyloxyethanol or 2-(2-ethoxyethoxy) ethanol. Organic solvents improve the cleaning performance but, for environmental reasons, are preferably not used. The organic solvent if present is used at a concentration of 1 - 5 wt%.

25 [0007] A fragrance is optionally present in the carpet cleaning composition. The fragrance is preferably an essential oil selected from lavender oil, deerhorn cedar oil, pine oil, orange oil and lemon oil used at a concentration of 0.5 - 1 wt.%.

Example

[0008] A carpet cleaning composition was prepared having the following composition:

- 5 10 wt.% of sodium alkyl sulphate with 14 carbon atoms
- 1 wt.% of Detnon (a commercial non-ionic surfactant)
- 2 wt.% of NoDirt
- 1.2 wt.% of Na-EDTA
- 0.5 wt% of a fragrance
- 10 and balance water

The fragrance used was lemon oil.

Comparative Example:

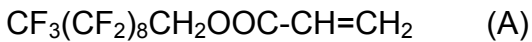
- 15 [0009] A comparative carpet cleaning composition was prepared which was identical to that employed in the example except that NoDirt was replaced by polyacrylic acid (PA JM45), which has been used in previous carpet cleaning compositions.

- 20 [0010] Two pieces of a pale coloured test carpet were provided. One piece was cleaned with the composition of the example. The other piece was cleaned with the composition of the comparative example. The carpets were allowed to dry for four hours and were then vacuum cleaned. A mixture of water, oil, mud, coffee and house dust was applied to both carpets. The mixture was worked into each carpet and then was allowed to dry
- 25 overnight. The carpets were then vacuum cleaned.

[0011] The degree of staining and soiling was evaluated visually. The carpets were between 1 (poor) and 10 (perfect) for their anti-staining and anti-soiling properties. The carpet cleaned with the composition of the invention had a value of 8. The carpet cleaned with the comparative composition had a rating of 6. The example demonstrates the effect of using the polyacrylate of the invention in carpet cleaning compositions.

Claims

[0012] 1. An aqueous carpet cleaning composition containing at least 5 wt.% surfactants, a sequestering agent, and 1-8 wt.% of a polyacrylate containing monomers of the formula A,



in which the monomers of the formula A constitute at least 20 wt.% of the monomers in the polyacrylate.

[0013] 2. The composition of claim 1 in which the polyacrylate contains 50 wt.% of monomers of formula A and 50 wt.% acrylic acid monomers.

[0014] 3. The composition of claim 1 which contains 2-3 wt.% of the polyacrylate.

Annex 4 (Document 2)

Journal of Allergies and Acarology: Brief Communications:

5 [0001] Dust mites (*Dermatophagoides* sp.) are one of the most important sources of allergens in modern houses. It would be very desirable to be able to reduce the number of dust mites in the home. The population of dust mites in mattresses and pillows can be minimised by the use of special covers. This approach is not possible for carpets and rugs. Studies have shown that benzyl benzoate and phenyl salicylate are acaricides and
10 can kill dust mites in carpets. The formulations used in these studies consisted of a solution of the compound in an organic solvent (typically a 1 wt.% solution in a glycol ether solvent) which was sprayed onto the carpet and left to dry. It is however necessary to use a very dilute solution containing a high concentration of organic solvent to ensure that the acaricide can be uniformly sprayed onto the carpet. The use of high
15 concentrations of organic solvents in the home is neither safe nor environmentally acceptable.

[0002] It has been investigated whether acaricides can be incorporated into carpet shampoos. Carpet shampoos are aqueous compositions which typically contain
20 surfactants, resoiling inhibitors, sequestering agents and a fragrance as well as water. A commercially successful carpet shampoo is disclosed in document 1.

[0003] Benzyl benzoate and phenyl salicylate were each dissolved in
25 2-(2-ethoxyethoxy)ethanol (a glycol ether solvent) at a weight ratio of acaricide: solvent of 1:2. These solutions were then stirred into a series of 5 different commercial carpet shampoos so as to give an acaricide concentration of 1 wt.% in the shampoo. The shampoo remained a clear liquid after the addition of the acaricide.

[0004] A 6-month study was performed in 100 homes. Each carpet shampoo was used in 20 homes of which 10 used the shampoo without acaricide, 5 homes used the shampoo containing the phenyl salicylate solution and 5 homes used the shampoo containing benzyl benzoate solution. The carpets were shampooed at the beginning of the study and again after 3 months using a rotary brush carpet cleaner, allowed to dry for 4 hours and then vacuum cleaned. The carpets were additionally vacuum cleaned weekly.

[0005] Dust samples were collected monthly from each carpet and the number of live dust mites in a 50 mg dust sample were counted. The sampling and measurement protocol used is that set out in J. Acar. Res., 46 (1985), 101. The cleaning performance of the shampoos was evaluated visually.

[0006] The average number of live dust mites in the carpets treated with a carpet shampoo containing an acaricide was 10 dust mites/g of dust. The carpets treated with the carpet shampoos which did not contain an acaricide contained on average 130 dust mites/g of dust. The presence of the acaricide was observed to have no effect on the cleaning performance of the carpet shampoos.

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Claims of Annex 1 (Patent application)

[0019] 1. A carpet shampoo consisting of:

5-20 wt.% of surfactant

1-8 wt.% of a resoiling inhibitor

0.1-5 wt.% of an acaricide

1-2 wt.% of a sequestering agent

0-5 wt.% of an organic solvent

and balance water

[0020] 2. The carpet shampoo of claim 1 where the surfactant is a mixture of at least one anionic and at least one non-ionic surfactant.

[0021] 3. The carpet shampoo of claim 1 where the surfactant mixture contains at least 50 wt.% of anionic surfactants.

[0022] 4. The carpet shampoo of claim 1 where the resoiling inhibitor is a fluorinated polyacrylate, containing monomer units of formula (I):



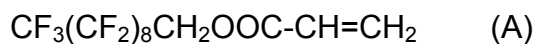
[0023] 5. The carpet shampoo of claim 1 where the acaricide is selected from benzyl benzoate and phenyl salicylate and the composition contains an organic solvent.

[0024] 6. The carpet shampoo of claim 5 where the organic solvent is a glycol ether.

[0025] 7. The carpet shampoo of claim 3 where the acaricide is selected from the essential oils thyme oil, deerhorn cedar oil, lemon oil and rosemary oil and contains no organic solvent.

Claims of Annex 3 (Document 1)

[0012] 1. An aqueous carpet cleaning composition containing at least 5 wt.% surfactants, a sequestering agent, and 1-8 wt.% of a polyacrylate containing monomers of the formula A,



in which the monomers of the formula A constitute at least 20 wt.% of the monomers in the polyacrylate.

[0013] 2. The composition of claim 1 in which the polyacrylate contains 50 wt.% of monomers of formula A and 50 wt.% acrylic acid monomers.

[0014]3. The composition of claim 1 which contains 2-3 wt.% of the polyacrylate.