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## (54) FABRIC TREATMENT COMPOSITIONS

- (57) A fabric treatment composition comprising by weight of the composition
- from 0.01 % to 5% of sulfonated tannins;
- from 0.1% to 50% of surfactant selected from anionic, cationic, nonionic surfactant, and mixtures thereof;
- from 0.1 % to 7% of dispersed perfume.

A use of the fabric treatment composition to improve freshness of fabrics treated with said composition.

A process of making the fabric treatment composition comprising the steps:

- a) combining one or more sulfonated tannins with an organic solvent to form a premix;
- b) combining said premix with the additional ingredients according to any preceding claim.

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#### Description

#### FIELD OF INVENTION

<sup>5</sup> **[0001]** The invention relates to fabric treatment compositions comprising sulfonated tannins, surfactants, and perfume. The invention further relates to a process of making and using same.

#### BACKGROUND OF THE INVENTION

[0002] Fabric treatment compositions used in the laundry process provide benefits to fabrics such as providing a pleasant smell. Such pleasant smell is typically provided by perfumes. A problem in the field is that much of the perfume is either not deposited or rinsed away during fabric treatment. Because perfumes are expensive components, encapsulation can be used in order to improve the delivery of the perfume during use. Unfortunately, encapsulation processes are time consuming and expensive as they typically require chemical reactions such as extensive crosslinking and/or result in capsules that over-protect the perfume as such capsules typically require a high energy input to release their active - for example pressure, temperature and/or electromagnetic radiation. Furthermore, such encapsulates' shell typically only protects the benefit agent and, on its own, provides no active value and may even leave a residue when a product containing the encapsulate is used. Deposition aids can also be used but typically only improve the deposition of a single specific perfume raw material.

[0003] Hence a need remains for fabric treatment compositions comprising a perfume and a deposition aid to improve the efficiency of deposition and/or retention of perfume onto fabrics, and/or improve release of perfume from said treated fabrics.

**[0004]** What is furthermore desired is a deposition aid which can be processed into the fabric treatment composition even when little energy is provided to the system, for example when only low shear mixing, needed to ensure homogeneous distribution of the perfume deposition aid throughout the composition, is provided. Such fabric treatment compositions comprising perfume deposition aid and methods of making and using same are provided herein.

[0005] WO2017/0088798 relates to cleaning compositions comprising tannins to mitigate malodors without imparting color to an article.

## 30 SUMMARY OF THE INVENTION

**[0006]** The invention relates to fabric treatment compositions comprising from 0.01% to 5% sulfonated tannins, from 0.1% to 7% of dispersed perfume and from 0.1% to 50% of surfactants, and processes for making and using same. Such treatment compositions provide improved perfume deposition and/or release from treated fabrics. In addition, such compositions are easy to process as a result of improved solubility of the sulfonated tannins into the treatment composition.

## DETAILED DESCRIPTION OF THE INVENTION

## Definitions

Definition 40

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[0007] As used herein, the term "fabric treatment composition" is a subset of cleaning and treatment compositions that includes, unless otherwise indicated, granular or powder-form all-purpose or "heavy-duty" washing agents, especially cleaning detergents; liquid, gel or paste-form all-purpose washing agents, especially the so-called heavy-duty liquid types; liquid fine-fabric detergents; liquid cleaning and disinfecting agents, fabric conditioning products including softening and/or freshening that may be in liquid, solid and/or dryer sheet form; as well as cleaning auxiliaries such as bleach additives and "stain-stick" or pre-treat types, substrate-laden products such as dryer added sheets, dry and wetted wipes and pads, nonwoven substrates, and sponges; as well as sprays and mists. All of such products which are applicable may be in standard, concentrated or even highly concentrated form even to the extent that such products may in certain aspect be non-aqueous.

[0008] As used herein, articles such as "a" and "an" when used in a claim, are understood to mean one or more of what is claimed or described.

[0009] As used herein, the terms "include", "includes" and "including" are meant to be nonlimiting.

[0010] As used herein, the term "solid" includes granular, powder, bar and tablet product forms.

[0011] As used herein, the term "fluid" includes liquid, gel, paste and gas product forms.

**[0012]** Unless otherwise noted, all component or composition levels are in reference to the active portion of that component or composition, and are exclusive of impurities, for example, residual solvents or by-products, which may be present in commercially available sources of such components or compositions.

[0013] All percentages and ratios are calculated by weight unless otherwise indicated. All percentages and ratios are

calculated based on the total composition unless otherwise indicated.

[0014] It should be understood that every maximum numerical limitation given throughout this specification includes every lower numerical limitation, as if such lower numerical limitations were expressly written herein. Every minimum numerical limitation given throughout this specification will include every higher numerical limitation, as if such higher numerical limitations were expressly written herein. Every numerical range given throughout this specification will include every narrower numerical range that falls within such broader numerical range, as if such narrower numerical ranges were all expressly written herein.

## Sulfonated tannins

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[0015] The fabric treatment composition comprises from 0.01% to 5%, preferably from 0.02% to 3%, more preferably from 0.03% to 1.5%, most preferably from 0.05% to 0.5% of sulfonated tannins. With "sulfonated tannins" we herein mean tannins comprising a sulfite (-SO<sub>3</sub>-) functional group. It was surprisingly found that sulfonated tannins improve the deposition of perfume onto fabrics and hence improve freshness of fabrics treated with the fabric treatment compositions according to the present invention. As such, sulphonated tannins act as perfume deposition aids. Furthermore, without wishing to be bound by theory, the sulfite functional group of sulfonated tannins enable said tannins to be easily processed in an aqueous intermediate and/or fabric treatment composition in case said fabric treatment composition comprises water. As such, sulfonated tannins reduce manufacturing complexity of fabric treatment compositions.

**[0016]** Tannins are polyphenolic secondary metabolites of higher plants, and are either galloyl esters and their derivatives, in which galloyl moieties or their derivatives are attached to a variety of polyol-, catechin- and triterpenoid cores (gallotannis, ellagitannins and complex tannins), or they are oligomeric and polymeric proanthocyanidis that can possess interflavanyl coupling and substitution patterns (condensed tannins). More details on the classification of tannins can be found in K. Khanbabaee, T. van Ree, Tannins: Classification and Definition, The Royal Society of Chemistry 2001, pages 641-649.

[0017] Gallotannins are all those tannins in which galloyl units are bound to diverse polyol-, catechin- or triterpenoid units.

[0018] Ellagitannins are those tannins in which at least two galloyl units are C-C coupled to each other, and do not contain a glycosidically linked catechin unit.

[0019] Complex tannins are tannins in which a catechin unit is bound glycosidically to a gallotannin or an ellagitannin unit.

**[0020]** Condensed tannins are all oligomeric and polymeric proanthocyanidins formed by linkage of C-4 of one catechin with C-8 or C-6 of the next monomeric catechin.

[0021] The table below provides classes of tannins more widely found in common higher plants.

COMMON NAME	FAMILY	GENUS	SPECIES (example of species studied)	STRUCTURE (tannins classes present at higher percentage in the plant extract)
Nutgall tree (sumac)	Anacardiacee	Rhus		Gallotannins Ellagitannins
Willow leaf	Anacardiacee	Shinopsis	balansae	Condensed
Red Quebracho	Anacardiacee	Shinopsis	lorentzii	Condensed
Grape seeds	Vitaceae	Vitis	vinifera	Condensed
Mimosa bark	Fabaceae Mimosoideae	acacia	mollissima	Condensed
(black wattle)	Fabaceae Mimosoideae	Acacia	mearnsii	Condensed
Quechua	Fabaceae	Caesalpinia sp.	spinosa	Gallotannins Ellagitannins
	Fabaceae	Sesbania		Condensed
trefoil	Fabaceae	Lotus		Condensed
sainfoin	Fabaceae	Onobrychis sp.		Condensed
	Fabaceae	Vicia	faba	Condensed

(continued)

COMMON NAME	FAMILY	GENUS	SPECIES (example of species studied)	STRUCTURE (tannins classes present at higher percentage in the plant extract)
oak	Fagaceae	Quercus sp.		Gallotannins Ellagitannins
chestnut	Fagaceae	Castanea	sativa	Ellagitannins
	Fagaceae Beech	Lithocarpus glaber sp.		Condensed
oak	Fagaceae	Quercus		Gallotannins Ellagitannins
maple	Sapindaceae	Acer		Gallotannins Ellagitannins
Pine bark	Pinaceae Pinoidaea	Pinus		Condensed
Spruce bark	Pinaceae	Picea		Condensed
		Sorghum		Condensed
	Rhizophoraceae	mangrove		Condensed
	Myrtaceae	Eucalyptus		Ellagitannins Gallotannins Condensed
Myrtan or black marlock	Myrtaceae	Eucalyptus	redunca	Condensed
Myrtle	Myrtaceae	Mirtus		Condensed
birch	Betulaceae	Betula		Gallotannins Ellagitannins
myrabolan	Combretaceae	Terminalia	chebula	Ellagitannins
	Rosaceae	Prunus sp.		Condensed
	Rosaceae	Malus sp.		Condensed
betel	Arecaceae	Areca	catechu	Condensed
	Burseraceae	Commiphora		Condensed
	Burseraceae	Angolensissp.		Condensed
	Burseraceae	Canarium sp.		Condensed
Persommon	Ebenaceae	Diospyros		Complex

**[0022]** Sulfonated tannins can be prepared using different processes. One suitable process to obtain sulfonated tannins is described in the EXAMPLES.

[0023] Preferably, said treatment composition comprises sulfonated tannins wherein said tannins are selected from the group consisting of gallotannins, ellagitannins, condensed tannins, complex tannins and mixtures thereof. Preferably, said gallotannins are extracted from sumac galls, Aleppo oak galls, or sumac leaves, more preferably said gallotannins are selected from the group consisting of tannins extracted from Aleppo oak galls. Preferably, said ellagitannins are extracted from chestnut bark, and chestnut wood, more preferably said ellagitannins are selected from the group consisting of tannins extracted from chestnut bark. Preferably, said condensed tannins are selected from the group consisting of tannins extracted from bark pine, querbracho, mimosa bark, spruce bark, grape seeds, more preferably said condensed tannins are selected from the group consisting of tannins extracted from bark pine and querbracho. Preferably said complex tannins are selected from the group consisting of tannins extracted from persimmon and tea leaves.

**[0024]** More preferably, said treatment composition comprises sulfonated tannins wherein said tannins are selected from the group consisting of condensed tannins, even more preferably said tannins are selected from the group consisting of tannins extracted from bark pine or querbracho.

## Fabric treatment composition

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**[0025]** The fabric treatment composition comprises, by weight of the composition, from 0.01% to 5% of sulfonated tannins; from 0.1% to 50% of surfactant selected from anionic, cationic, nonionic surfactant, and mixtures thereof; from 0.1% to 7% of dispersed perfume.

[0026] Preferably, the fabric treatment composition comprises dispersed perfume at a level of from 0.2% to 5%, more preferably from 0.3% to 3.5%, more preferably 0.4% to 3%, most preferably from 0.5% to 2.5% by weight of the fabric treatment composition. By "dispersed perfume" we herein mean a perfume composition that is freely dispersed in the fabric treatment composition and is not encapsulated. Perfume is typically added to provide the fabric treatment composition with a pleasant smell. A perfume composition comprises one or more perfume raw materials (PRM). Perfume raw materials are the individual chemical compounds that are used to make a perfume composition. The choice of type and number of perfume raw materials is dependent upon the final desired scent. In the context of the present invention, any suitable perfume composition may be used. Those skilled in the art will recognize suitable compatible perfume raw materials for use in the perfume composition, and will know how to select combinations of ingredients to achieve desired scents.

**[0027]** The perfume composition may comprise from 2.5% to 30%, preferably from 5% to 30% by total weight of perfume composition of perfume raw materials characterized by a logP lower than 3.0, and a boiling point lower than 250°C.

[0028] The perfume composition may comprise from 5% to 30%, preferably from 7% to 25% by total weight of perfume composition of perfume raw materials characterized by having a logP lower than 3.0 and a boiling point higher than 250°C. The perfume composition may comprise from 35% to 60%, preferably from 40% to 55% by total weight of perfume composition of perfume raw materials characterized by having a logP higher than 3.0 and a boiling point lower than 250°C. The perfume composition may comprise from 10% to 45%, preferably from 12% to 40% by total weight of perfume composition of perfume raw materials characterized by having a logP higher than 3.0 and a boiling point higher than 250°C.

**[0029]** Preferred fabric treatment composition comprise dispersed perfume wherein said dispersed perfume comprises perfume raw materials selected from the list consisting of Hexyl salicylate, Hexyl cinnamic aldehyde, Verdox®, Iso E super®, and mixtures thereof.

**[0030]** Preferably said fabric treatment composition is a liquid fabric treatment composition. It is furthermore preferred that that said fabric treatment composition comprises, from 1% to 97%, preferably from 5% to 90%, more preferably from 8% to 80% of water.

**[0031]** In one aspect, said fabric treatment composition contains, based on total composition weight, 0.1 wt % to 50 wt %, advantageously 1 to 40 wt %, 3 to 30 wt % or 5 wt % to 20 wt % surfactant selected from the groups of anionic surfactants, nonionic surfactants, cationic, zwitterionic and/or amphoteric surfactants. However, it is particularly preferred if the fabric treatment composition according to the invention contains anionic, nonionic and/or cationic surfactants. This corresponds to a preferred embodiment of the invention and enables optimum cleaning and/or softness performance.

[0032] In one aspect, said fabric treatment composition contains, based on total composition weight, from 0.1 to 80 wt %, 1 to 60 wt %, or 5 to 50 wt % builders.

[0033] In one aspect, said fabric treatment composition contains a soluble builder system, in one aspect, said soluble builder system comprises soda, silicate, citrate and/or polycarboxylates.

**[0034]** In one aspect, said fabric treatment composition contains a perfume and a perfume delivery system. This perfume delivery system comprises a material selected from the group consisting of microcapsules, a polymer assisted delivery system; a molecule-assisted delivery system; a fiber-assisted delivery system; a cyclodextrin delivery system; a starch encapsulated accord; and/or an inorganic carrier delivery system.

**[0035]** In one aspect, said perfume microcapsule comprises a material selected from the group consisting of melamine-formaldehyde polymers, melamine-urea polymers, polyurea, polyurethane, polyacrylate, polymethylmethacylate and polyacrylate esters.

[0036] In one aspect, said microcapsules contain aromatic substances.

**[0037]** In one aspect, said benefit agent delivery particles may have any combination of the aforementioned parameters as listed in the aforementioned aspects.

**[0038]** It is particularly preferred if the fabric treatment composition further comprises an organic solvent, preferably said organic solvent is an alcohol, more preferably said organic solvent is 1,2-propane diol. In one aspect, a process for manufacturing a fabric treatment composition, comprises premixing sulfonated tannins with an organic solvent prior to manufacturing the final fabric treatment composition. Preferably said organic solvent is an alcohol, more preferably said organic solvent is 1,2-propane diol.

[0039] The tannins are stable within the fabric treatment composition. During use of the fabric treatment composition, for example when washing or rinsing laundry, the sulfonated tannins improve the deposition of perfume raw materials and as such provide freshness benefits.

[0040] It is particularly preferred if the fabric treatment composition contains anionic surfactant, advantageously in

amounts from 0.1 to 25 wt%, more advantageously 1 to 20 wt%, and in particular in amounts of 3 to 15 wt%, based on the product as a whole. This corresponds to a preferred embodiment of the invention and enables particularly advantageous cleaning performance. One particularly suitable anionic surfactant is alkyl benzene sulfonate, preferably linear alkyl benzene sulfonate (LAS). If the composition according to the invention contains alkyl benzene sulfonate, advantageously in amounts of 0.1 to 25 wt%, more advantageously 1 to 20 wt%, and in particular in amounts of 3 to 15 wt%, based on the product as a whole, this constitutes a preferred embodiment of the invention.

[0041] Other particularly suitable anionic surfactants are alkyl sulfates, in particular fatty alcohol sulfates (FAS) such as, for example,  $C_{12}$ - $C_{18}$  fatty alcohol sulfate. Cs-Cis alkyl sulfates can preferably be used; particularly preferred are  $C_{13}$  alkyl sulfate and  $C_{13}$ - $C_{15}$  alkyl sulfate and  $C_{13}$ - $C_{17}$  alkyl sulfate, advantageously branched, in particular alkyl-branched  $C_{13}$ - $C_{17}$  alkyl sulfate. Particularly suitable fatty alcohol sulfates can be derived from lauryl and myristyl alcohol; i.e. fatty alcohol sulfates containing 12 or 14 carbon atoms. Long-chained FAS types ( $C_{16}$  to  $C_{18}$ ) are very suitable for washing laundry at higher temperatures. Other preferred anionic surfactants that can be used include alkane sulfonates (e.g. secondary  $C_{13}$ - $C_{18}$  alkane sulfonate), methyl ester sulfonates (e.g.  $C_{12}$ - $C_{18}$  methyl ester sulfonate) and  $\alpha$ -olefin sulfonates (e.g.  $C_{12}$ - $C_{18}$  fatty alcohol-2EO ether sulfate) and/or soaps. Other suitable anionic surfactants will be described further below. However, particularly suitable are FAS and/or LAS.

**[0042]** Anionic surfactants, including the soaps, can be in the form of their sodium, potassium or ammonium salts, as well as soluble salts of organic bases such as mono-, di- or triethanolamine. Preferably, the anionic surfactants are present in the form of their sodium or potassium salts, in particular in the form of sodium salts.

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[0043] It is particularly preferred if the fabric treatment composition according to the invention contains nonionic surfactants, advantageously in amounts of 3 to 15 wt %, more advantageously 1 to 20 wt %, and in particular in amounts of 3 to 15 wt %, based on the product as a whole. This corresponds to one preferred embodiment of the invention. Particularly preferred is the use of alkyl polyglycol ethers, in particular in combination with anionic surfactant, such as, preferably, LAS. Other suitable nonionic surfactants are alkyl phenol polyglycol ether (APEO), (ethoxylated) sorbitan fatty acid ester (sorbitans), alkyl polyglucosides (APG), fatty acid glucamides, fatty acid ethoxylates, amine oxides, ethylene oxide propylene oxide block polymers, polyglycerol fatty acid ester, and/or fatty acid alkanol amides. Other suitable nonionic surfactants will be described further below. Sugar-based nonionic surfactants, such as, in particular, APG, are particularly preferred.

[0044] For the purposes of the invention, builders include in particular zeolites, polycarboxylates, citrates (such as, for example, sodium citrate, soda, sodium hydrogen carbonate, phosphates, sodium silicates (soluble glass), phosphonates, alkaline amorphous disilicates, and crystalline layered silicates. Builders are contained in fabric treatment compositions according to the invention preferably in amounts of 0.1 to 80 wt %, advantageously 1 to 60 wt %, and more advantageously 5 to 50 wt %. In addition, it is most particularly preferred that the detergent or cleaning agent according to the invention contain a builder system (i.e. at least two substances having a builder effect), preferably a builder system containing zeolite, preferably comprising zeolite in amounts > 1 wt %, advantageously > 5 wt %, more advantageously > 10 wt %, in particular ≥ 15 wt %, wt % based on the product as a whole. A useful maximum amount can be 40 wt %, 30 wt % or 20 wt %, based on the product as a whole. This corresponds to a preferred embodiment of the invention. A combination of zeolite and soda is preferred.

**[0045]** It is also particularly preferred if the fabric treatment composition according to the invention contains a soluble builder system, preferably comprising soda, silicate, citrate and/or polycarboxylates, advantageously in amounts of 0.1 to 50 wt %, based on the product as a whole. This corresponds to a preferred embodiment of the invention. If such a soluble builder system is contained in the product, it is most preferable if the product contains only minor amounts of insoluble builders, such as, in particular, zeolite, for example < 5 wt % to 0.1 wt %, and in particular, if the product in such cases contains no insoluble builder at all.

**[0046]** It is also particularly preferred if the fabric treatment composition according to the invention further comprises a chelant and/or an antioxidant, preferably said composition comprises a chelant. Chelants and antioxidants further improve the stability of perfume and as such, the perfume character is maintained over a longer time.

**[0047]** It is also possible for the fabric treatment composition according to the invention to contain phosphates. Phosphate is preferably contained in amounts of 1 to 40 wt %, in particular 5 to 30 wt %, based on the product as a whole. However, according to another preferred embodiment, the detergent or cleaning agent according to the invention is free of phosphates.

**[0048]** The fabric treatment composition according to the invention, which, for example, can be present as, in particular, solids in powder form, in passivated particle form, as homogenous solutions or suspensions, can also in principle contain all known ingredients that are customary in such products. The products according to the invention can, as was already shown, contain in particular builder substances, surfactants, also bleaching agents, bleach activators, water-miscible organic solvents, enzymes, sequestering agents, electrolytes, pH regulators, and other additives such as optical brighteners, fluorescing agents, anti-redeposition agents, shrinkage blockers, anti-creasing agents, color-transfer inhibitors, antimicrobial active substances, germicides, fungicides, antioxidants, preservatives, corrosion inhibitors, glass corrosion inhibitors, disintegrating agents, static inhibitors, bitters, ironing aids, water-repellent and impregnating agents, swelling

and anti-slip agents, neutral filling salts, as well as UV absorbers, foam regulators, as well as colorants and aromatic substances.

**[0049]** In one aspect of the invention, the fabric treatment composition comprises cationic surfactant. Suitable cationic surfactants include quaternary ammonium ester softening actives. Said suitable quaternary ammonium ester softening actives include, but are not limited to, materials selected from the group consisting of monoester quats, diester quats, triester quats and mixtures thereof. Preferably, the level of monoester quat is from 2.0% to 40.0%, the level of diester quat is from 40.0% to 98.0%, the level of triester quat is from 0.0% to 25.0% by weight of total quaternary ammonium ester softening active.

[0050] Said quaternary ammonium ester softening active may comprise compounds of the following formula:

$$\{R_{(4-m)}^2 - N + - [X - Y - R_1]_m\} A$$

wherein:

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m is 1, 2 or 3 with proviso that the value of each m is identical;

each R<sup>1</sup> is independently hydrocarbyl, or branched hydrocarbyl group, preferably R<sup>1</sup> is linear, more preferably R<sup>1</sup> is partially unsaturated linear alkyl chain;

each  $R^2$  is independently a  $C_1$ - $C_3$  alkyl or hydroxyalkyl group, preferably  $R^2$  is selected from methyl, ethyl, propyl, hydroxyethyl, 2-hydroxypropyl, 1-methyl-2-hydroxyethyl, poly( $C_{2^-3}$  alkoxy), polyethoxy, benzyl;

each X is independently -(CH $_2$ ) $_n$ -, -CH $_2$ -CH(CH $_3$ )- or -CH(CH $_3$ )-CH $_2$ - and

each n is independently 1, 2, 3 or 4, preferably each n is 2;

each Y is independently -O-(O)C- or -C(O)-O-;

A- is independently selected from the group consisting of chloride, methyl sulfate, and ethyl sulfate, preferably A- is selected from the group consisting of chloride and methyl sulfate;

with the proviso that when Y is -O-(O)C-, the sum of carbons in each R<sup>1</sup> is from 13 to 21, preferably from 13 to 19. Preferably, X is -CH<sub>2</sub>-CH(CH<sub>3</sub>)- or -CH(CH<sub>3</sub>)-CH<sub>2</sub>- to further improve the hydrolytic stability of the quaternary ammonium ester softening active, and hence further improve the stability of the fabric treatment composition.

**[0051]** Because of the balance of processability and odor of the quaternary ammonium ester softening active, in preferred fabric treatment compositions, the iodine value of the parent fatty acid from which the quaternary ammonium fabric softening active is formed is from 0 to 100, more preferably from 10 to 60, even more preferably from 15 to 45.

**[0052]** Examples of suitable quaternary ammonium ester softening actives are commercially available from KAO Chemicals under the trade name Tetranyl® AT-1 and Tetranyl® AT-7590, from Evonik under the tradename Rewoquat® WE16 DPG, Rewoquat® WE18, Rewoquat® WE20, Rewoquat® WE28, and Rewoquat® 38 DPG, from Stepan under the tradename Stepantex® GA90, Stepantex® VR90, Stepantex® VK90, Stepantex® VA90, Stepantex® DC90, and Stepantex® VL90A.

[0053] A process of making fabric treatment composition according to the present invention comprises the steps:

a. combining one or more sulfonated tannins with an organic solvent to form a premix, preferably said organic solvent is an alcohol, more preferably said organic solvent is 1,2-propane diol;

b. combining said premix with the additional ingredients according to any preceding claim.

**METHODS** 

Washing procedure - Launder-o-Meter test

**[0054]** The washing procedure in small scale washing setup (Roaches® Washtec Launder-o-Meter) comprised different steps:

- 1) A 5 cm x 5 cm piece of cotton fabric is weighed (0.8 g) and put into the metal Launder-o-Meter jar;
- 2) 10 metal spheres (stainless steel, mass 2 g, diameter 7 mm) are added into the jar to facilitate mixing;
- 3) 200 mL of demineralized water is added to the jar;
- 4) Unless otherwise mentioned, 1.0-1.1 g of liquid detergent composition containing the fabric treatment composition is added into the jar;
- 5) The jar is closed and placed into the Lauder-o-Meter;
  - 6) The temperature is controlled to 37-38°C;
  - 7) After 1 hour, the jar is removed from the Launder-o-Meter;
  - 8) The wet fabrics are squeezed to remove excess water by applying the same pressure to obtain the same weight

of the different wet fabrics;

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- 9) The jar and the metal spheres are rinsed with demineralized water;
- 10) The treated fabric is put again into the jar together with the metal spheres;
- 11) 200 mL of demineralized water are added to the jars;
- 12) The jars are closed and placed into the Launder-o-Meter;
- 13) The temperature is controlled to be 37-38°C;
- 14) After 5 minutes, the jar is removed from the Launder-o-Meter;
- 15) The fabric is squeezed by applying the same pressure to obtain the same wet weight;
- 16) The wet fabric is placed into a vial for GC-MS head space analysis and the vial is closed.

Headspace analysis through Gas Chromatography - Mass Spectroscopy (GC-MS) - Lauder-o-Meter test

[0055] For each fabric sample a chromatogram is recorded and 3 measurements for each sample are performed.

**[0056]** The obtained correlation areas are related to the single PRM concentrations, expressed in nM. The concentrations of the most abundant PRMs are taken into account: Hexyl salicylate; Hexyl Cinnamic aldehyde; Verdox; and Iso E super.

**[0057]** The headspace concentration is expressed relative to a comparative reference according according to the below equation:

 $Relative \ difference \ \% = \left(1 - \frac{PRM \ concentration \ of \ comparative \ sample}{PRM \ concentration \ of \ inventive \ sample}\right) * \ 100\%$ 

**[0058]** This procedure is repeated for each most abundant PRM in the perfume accord tested. The same calculation is repeated for the second and the third measurements and the average value of relative difference is calculated.

Full scale washing machine test

**[0059]** The washing procedure in full scale is performed by using a Miele W1714 washing machine operating at 30 °C, 800 rpm and synthetic short program with 2 gpg water. Cotton and polyester fabric tracers (20 cm x 20 cm) are put into the washing machine together with a mixed cotton/polycotton ballast load (8 cotton pieces of 134 g each and 8 polycotton pieces of 124 g each). Fabric treatment compositions are added into the washing machine together with the washing load.

Deposition test through Headspace analysis via Gas Chromatography - Mass Spectroscopy (GC-MS) - Washing machine test

**[0060]** Wet fabrics coming from the washing machine are dried at room temperature for 15 hours and then cut into 5 cm x 5 cm pieces. Samples for deposition tests are prepared as follows:

- 1) Each dried piece of treated fabric is put into a 20 mL vial;
- 2) 12 mL of ethanol are poured into each vial containing the fabrics;
- 3) 12 mL of ethanol are also poured into 3 vials for the preparation of the standards (at 3 different perfume levels);
- 4) All the vials containing the fabrics are placed into an oven with orbital shaking at 60 °C for 1 hour;
- 5) All the vials are transferred into an ultrasonic bath and left under sonication for 15 minutes;
- 6) The vials are shaken and each ethanol solution is poured into a different vial (containing the extracts);
- 7) A stock PRM solution (0.5% w/w of PRM in ethanol) is prepared to make a work solution with a concentration of 0.008% w/w of PRM in ethanol; standard solutions are prepared by pouring 3 different amounts or work solutions are added to the vials containing 12 mL of ethanol;
- 8) 4.5 mL of a 20% w/w solution of NaCl are transferred into a vial for GC-MS head-space analysis. This procedure is repeated for each extract and standard solution. Each sample is replicated;
- 9) 0.5 mL of PRM solutions in ethanol (extracts and standards) are poured into the vials containing the NaCl solution;
- 10) The vials are closed and the GC-MS analysis is started.
- [0061] After the measurements, the obtained correlation areas are prepared.

#### **EXAMPLES**

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[0062] Sulfonated pine bark extract tannins were prepared as follows:

- 1. Ground Finland pine barks (57.0% H<sub>2</sub>O) were oven dried for 24h at 50°C.
- 2. 500 g of dried pine bark of step 1 (8.0%  $H_2O$ ) were placed with 5000 g of a 2.0% by weight homogenous aqueous solution of sodium disulfite ( $Na_2O_5S_2$ ) in a water heated 10L glass reactor equipped with a mechanical stirrer and a condenser.
- 3. The resulting mixture of step 2 was heated under stirring to 80°C and was kept to this temperature for 2h. The resulting mixture was then cooled to 40°C and filtered through a nylon gauze.
- 4. 2500 g of a 2.0% by weight of homogenous aqueous solution of sodium disulfite ( $Na_2O_5S_2$ ) were added to the filtered resulting mixture of step 3 in the same glass reactor. The resulting mixture was heated under stirring to 80°C and was kept to this temperature for 2h. The resulting mixture was then cooled to 40°C and filtered through nylon gauze.
- 5. Both filtrates of steps 3 and 4 were pooled together and filtered through a paper filter.
- 6. The filtrate of step 5 was concentrated in a 10 L glass balloon on a rotary evaporator at 80°C under a vacuum of 350 mbar up to a volume of 10 L. The mixture was then transferred to a 2 L glass balloon and concentrated to dryness under vacuum at 1 mbar and 80°C.
- 7. The extract of step 6 was scratched from the balloon and crushed into a mortar and 135.0 g of extract was recovered. The yield of extraction was 27.0%.
- 8. The sulfite content of the extract, measured by a spectrophotochemical method using Ellman's reagent (5-5'-dithiobis (2-nitrobenzoic acid)), was 51.8%.
- 9. The condensed tannins content, determined by the Scalbert method, was 22mgCyE/g (Scalbert, A., 1992. Quantitative methods for the estimation of tannins in plant tissues. In: Hemingway, R.W., Laks, P.E. (Eds), Plant Polyphenols: Synthesis, Properties, Significance. Plenum Press, New York).

[0063] Fabric treatment compositions were prepared by providing the following materials:

30	Material	Supplier
30	1,2 Propanediol	INEOS
	Perfume <sup>a</sup>	PROCTER & GAMBLE
	Unperfumed Liquid Detergent	PROCTER & GAMBLE
35	<sup>a</sup> Perfume accord comprising more than 20 PRM's including aldehyde, Verdox, and Iso E super.	most abundant PRM's Hexyl salicylate, Hexyl cinnamic

**[0064]** Sulfonated tannin solutions were prepared by adding the sulfonated pine bark extract tannins and 1,2 propanediol into a glass vial. In order to further accelerate the solubilization of the tannins, the solutions were put into an ultrasonic bath for 30 minutes. The fabric treatment compositions were prepared by adding perfume and, when present, the sulfonated tannins solutions to an unperfumed liquid detergent in a glass jar and mixing until a homogeneous fabric treatment composition was obtained.

Table 1: Composition of examples 1 to 4. Comparative example is indicated with an asterisk.

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	Ex. 1*	Ex. 2	Ex. 3	Ex. 4	
		W	eight %		
Water Balance to 100%					
Perfume	0.72%				
sulfonated pine bark extract tannins		0.05%	0.10%	0.20%	
C11.8 Linear Alkyl Benzene (HLAS)		6.90			
Amines, C12-14 (even numbered) -alkyldimethyl, N-oxides	0.660				

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(continued)

	Ex. 1*	Ex. 2	Ex. 3	Ex. 4
Poly(oxy-1,2-ethanediyl), alpha-sulfo-omega-hydroxy-, C10-16-alkyl ethers, sodium salts			11.2	
C12-C18 fatty acids			0.878	
Citric Acid			2.00	
DTPA			0.453	
Enzymes, stabilizers			1.25	
Aziridine, homopolymer, ethoxylated			1.51	
Aziridine, polymer with 2-methyloxirane and oxirane	1.32			
Brighteners	0.181			
Ethanol			1.44	
1,2 Propanediol			2.05	
Sorbitol	0.0630			
Monoethanolamine	2.09			
Diethylene glycol	1.93			
Sodium hydroxyde	0.676			
Anti-foam, aesthetic, dye	0.217			

**[0065]** Cotton fabrics were treated with the fabric treatment compositions Ex. 1 to Ex. 4 in a Launder-o-Meter washing test (see Methods). GC-MS headspace analysis was carried out on wet fabrics after washing fabric treatment. Table 2 summarizes the headspace results, which reflect the relative headspace concentration of the 5 most abundant PRM's above the wet treated fabrics. A higher headspace concentration difference above wet treated fabrics is indicative of improved deposition and/or release of perfume. The error on the measurements is the standard deviation from 3 independent headspace measures.

Table 2: Relative headspace concentration differences above wet fabrics treated with inventive compositions ex. 2 to 4 as compared to comparative composition ex. 1. Only the relative differences for the most abundant Perfume Raw Materials contained in the perfume accord are shown.

Perfume Raw Material	Relative difference %			
renume Naw Material	Ex. 2 vs Ex. 1	Ex. 3 vs Ex. 1	Ex. 4 vs Ex. 1	
Hexyl salicylate	+40 ± 5%	+51 ± 8%	+48 ± 9%	
Hexyl cinnamic aldehyde	+44 ± 7%	+53 ± 6%	+50 ± 5%	
Verdox	+29 ± 4%	+51 ± 1%	+39 ± 2%	
Iso E super	+34 ± 2%	+47 ± 1%	+39 ± 2%	

**[0066]** By comparing ex. 2, ex. 3 and ex. 4 with ex. 1, it is clear that the compositions according to the invention (ex. 2-4) resulted in a consistent significantly higher headspace concentration above treated wet fabrics indicating improved deposition and/or release of perfume.

**[0067]** Table 3 summarizes the composition of the samples used to treat fabrics according to the full scale washing machine test (see Methods), followed by GC-MS analysis to assess the deposition (see Methods).

Table 3: Composition of examples 5 and 6. Example 5 is a comparative example indicated with an asterisk.

Ex. 5*	Ex. 6	
Weight %		

(continued)

Water	Balance to 100%	
Perfume	0.72%	
sulfonated pine bark extract tannins	- 1%	
C11.8 Linear Alkyl Benzene (HLAS)	6.90	
Amines, C12-14 (even numbered) -alkyldimethyl, N-oxides	0.660	
Poly(oxy-1,2-ethanediyl), alpha-sulfo-omega-hydroxy-, C10-16-alkyl ethers, sodium salts	11.2	
C12-C18 fatty acids	0.878	
Citric Acid	2.00	
DTPA	0.453	
Enzymes, stabilizers	1.25	
Aziridine, homopolymer, ethoxylated	1.51	
Aziridine, polymer with 2-methyloxirane and oxirane	1.32	
Brighteners	0.181	
Ethanol	1.44	
1,2 Propanediol	2.05	
Sorbitol	0.063	
Monoethanolamine	2.09	
Diethylene glycol	1.93	
Sodium hydroxyde	0.676	
Anti-foam, aesthetic, dye	0.217	
	Perfume sulfonated pine bark extract tannins C11.8 Linear Alkyl Benzene (HLAS) Amines, C12-14 (even numbered) -alkyldimethyl, N-oxides Poly(oxy-1,2-ethanediyl), alpha-sulfo-omega-hydroxy-, C10-16-alkyl ethers, sodium salts C12-C18 fatty acids Citric Acid DTPA Enzymes, stabilizers Aziridine, homopolymer, ethoxylated Aziridine, polymer with 2-methyloxirane and oxirane Brighteners Ethanol 1,2 Propanediol Sorbitol Monoethanolamine Diethylene glycol Sodium hydroxyde	

**[0068]** Table 4 reports the total correlation areas obtained from the GC-MS analysis. A higher correlation area is indicative of higher total PRM content and as such is indicative of improved deposition of perfume on the treated fabrics.

Table 4: Correlation areas from deposition tests using compositions ex. 6 as compared to comparative composition

	Ex. 5*	Ex. 6
Correlation area x 10 <sup>-4</sup> [area counts]	61 ± 9	114 ± 8

**[0069]** By comparing ex. 6 with ex. 5, it is clear that the composition ex. 6, according to the present invention, resulted in a higher PRM deposition, since the values of the calculated correlation area are almost double as compared to those of comparative ex. 5.

## **Claims**

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- 1. A fabric treatment composition comprising by weight of the composition
  - from 0.01% to 5% of sulfonated tannins;
  - from 0.1% to 50% of surfactant selected from anionic, cationic, nonionic surfactant, and mixtures thereof;
  - from 0.1% to 7% of dispersed perfume.
- 2. The fabric treatment composition according to claim 1 wherein the level of said sulfonated tannins is from 0.02% to 3%, preferably from 0.03% to 1.5%, most preferably from 0.05% to 0.5%.

- 3. The fabric treatment composition according to any preceding claim wherein said tannins are selected from the group consisting of gallotannins, ellagitannins, condensed tannins, complex tannins, and mixtures thereof.
- 4. The fabric treatment composition according to claim 3 wherein

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- -said gallotannins are extracted from sumac galls, Aleppo oak galls, or sumac leaves, preferably said gallotannins are selected from the group consisting of tannins extracted from Aleppo oak galls;
- said ellagitannins are extracted from chestnut bark, and chestnut wood, preferably said ellagitannins are selected from the group consisting of tannins extracted from chestnut bark;
- said condensed tannins are selected from the group consisting of tannins extracted from bark pine, querbracho, mimosa bark, spruce bark, grape seeds, and mixtures thereof; preferably said condensed tannins are selected from the group consisting of tannins extracted from bark pine and querbracho;
- complex tannins are selected from the group consisting of tannins extracted from persimmon and tea leaves.
- 5. The fabric treatment composition according to claim 4 wherein said tannins are selected from the group consisting of condensed tannins, more preferably said tannins are selected from the group consisting of tannins extracted from bark pine or querbracho.
- 6. The fabric treatment composition according to any preceding wherein said fabric treatment composition is a liquid fabric treatment composition comprising from 1% to 97%, preferably from 5% to 90%, more preferably from 8% to 80% of water by weight of the fabric treatment composition.
  - 7. The fabric treatment composition according to any preceding claim wherein the level of dispersed perfume is from 0.2% to 5%, preferably from 0.3% to 3.5%, more preferably 0.4% to 3%, most preferably from 0.5% to 2.5% by weight of the fabric treatment composition.
  - 8. The fabric treatment composition according to any preceding claim wherein said dispersed perfume comprises a perfume raw material selected from the list consisting of Hexyl salicylate, Hexyl cinnamic aldehyde, Verdox, Iso E super, and mixtures thereof.
  - **9.** The fabric treatment composition according to any preceding claim wherein the surfactant is present at a level of from 1 to 40 %, preferably from 3 to 30 %, more preferably from 5 % to 20 %, by weight of the fabric treatment composition.
- 10. The fabric treatment composition according to any preceding claim wherein the surfactant is selected from anionic and nonionic surfactant, preferably said surfactant is selected from the list consisting of alkyl benzene sulfonate, fatty alcohol sulfates, alkyl polyglycol ethers, alkyl phenol polyglycol ether, sorbitan fatty acid ester, alkyl polyglucosides, fatty acid glucamides, fatty acid ethoxylates, amine oxides, ethylene oxide propylene oxide block polymers, polyglycerol fatty acid ester, and/or fatty acid alkanol amides, and mixtures thereof.
  - **11.** The fabric treatment composition according to any preceding claim wherein said composition further comprises a chelant and/or an antioxidant, preferably said composition comprises a chelant.
- **12.** The fabric treatment composition according to any preceding claim wherein said composition further comprises an organic solvent, preferably said organic solvent is an alcohol, more preferably said organic solvent is 1,2-propane diol.
  - **13.** A use of the fabric treatment composition according to any preceding claim to improve freshness of fabrics treated with said composition.
- 50 **14.** A process of making the fabric treatment composition according to any preceding claim comprising the steps:
  - a) combining one or more sulfonated tannins with an organic solvent to form a premix;
  - b) combining said premix with the additional ingredients according to any preceding claim.
- 15. The process according to claim 14 wherein the organic solvent is an alcohol, preferably said organic solvent is 1,2-propane diol.



## **EUROPEAN SEARCH REPORT**

Application Number EP 18 15 3131

	Citation of document with in	ndication, where appropriate,	B	elevant	CLASSIFICATION OF THE
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page 1 of 2



## **EUROPEAN SEARCH REPORT**

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CLASSIFICATION OF THE APPLICATION (IPC)

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