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(54) Title: LIQUID FABRIC CONDITIONING COMPOSITIONS

(57) Abstract: A liquid fabric conditioning composition is disclosed. The composition comprises 1 to 30% of a fabric conditioning agent, 0.1 to 30% of a capsule having an oil core and a capsule wall encapsulating the oil core, and 20 ppm or greater of a stabilizing agent. Also disclosed is a method of stabilizing the viscosity of a liquid fabric conditioning composition.



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LIQUID FABRIC CONDITIONING COMPOSITIONS

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims priority to US Patent Application, Serial No. 62/095,475, filed on December 22, 2014, the content of which is incorporated herein by reference in their entirety.

BACKGROUND

[0002] Liquid fabric conditioning compositions are widely used for providing fabric softening and static control benefits during laundering. They usually contain cationic surfactants, which forms multi-walled vesicles (similar to liposomes) responsible for viscosity of the liquid compositions.

[0003] In addition to surfactants, liquid fabric conditioning compositions also have small amount of other ingredients such as fragrance, salts for lowering the viscosity. During storage, these ingredients trigger changes vesicles, leading to significant increases in viscosity and rendering the conditioning composition not pourable or dispersable, and thus not usable. See US 5,447,644 and US 6,559,117. Further, as the concentration of the surfactant increases in the liquid fabric conditioning composition, the viscosity increases and tends to change more dramatically. In order to have a reasonable shelf life, current fabric conditioning compositions contain low concentrations of softening agents.

[0004] There is a need to develop a liquid fabric conditioning composition that has a prolonged viscosity stability and can accommodate a high concentration of a softening agent.

SUMMARY

[0005] The present invention is based on the discovery of a liquid fabric softening composition having an improved viscosity. The composition can also be formulated to have a high concentration of a softening agent.

[0006] One aspect of this invention relates to a liquid fabric conditioning composition comprising 1 to 30% (*e.g.*, 2 to 25% and 4 to 15%) of a fabric conditioning agent and 20 ppm or greater (*e.g.*, 50 ppm to 2%, 50 ppm to 1%, and 50 to 2000 ppm) of a stabilizing agent. The percentages are all by weight of the liquid fabric conditioning composition.

[0007] The stabilizing agent is a multi-functional amine, a mono-functional amine, an amino acid, a polymer mixture, or a combination thereof, the polymer mixture containing a first polymer selected from the group consisting of polyvinylpyrrolidone, polyvinylpyridine-N-oxide, polylysine, and polyvinyl imidazolium, and a second polymer.

[0008] Examples of a stabilizing agent include hexamethylene diamine, hexaethylenediamine, ethylenediamine, 1,3-diamino-propane, 1,4-diamino-butane, diethylenetriamine, pentaethylenehexamine, bis(3-amino-propyl)amine, bis(hexanethylene)triamine, tris(2-aminoethyl)amine, triethylene-tetramine, N,N'-bis(3-aminopropyl)-1,3-propanediamine, tetraethylenepentamine, branched polyethylenimine, chitosan, nisin, gelatin, 1,3-diamino-guanidine, 1,1-dimethylbiguanide, guanidine, arginine, lysine, ornithine, histidine, amino-2-methyl-1-propanol, and a combination thereof. Preferably, the polyfunctional amine is hexamethylene diamine, 1,3-diaminoguanidine, 1,1-dimethylbiguanide, guanidine, arginine, lysine, or a combination thereof. Preferably, the stabilizing agent is hexamethylenediamine, amino-2-methyl-1-propanol, lysine, arginine, histidine, or a combination thereof. Preferably, the stabilizing agent is hexamethylenediamine, amino-2-methyl-1-propanol, lysine, arginine, histidine, or a combination thereof. More preferably, the stabilizing agent is hexamethylenediamine.

[0009] The above fabric liquid conditioning composition can further contains 0.1 to 30% (*e.g.*, 0.15 to 15%, and 0.5 to 10%) of a capsule having an oil core and a capsule wall, the oil core containing an active material and the capsule wall encapsulating the oil core. The active material is a fragrance, a malodor counteractant, or a combination thereof. The capsule wall is formed of polyurea, polyurethane, polyacrylate, polyacrylamide, poly(acrylate-co-acrylamide), starch, silica, gelatin and gum Arabic, poly(melamine-formaldehyde), poly(urea-formaldehyde), or a combination thereof. Preferably, the capsule wall is formed of polyurea, polyurethane, or poly(melamine-formaldehyde).

[0010] The capsule typically contains 35 to 99.5% (*e.g.*, 50 to 98%) of the core oil and 0.5 to 65% (*e.g.*, 2 to 50%) of the capsule wall

[0011] The above liquid fabric conditioning composition can further contain a second, third, or fourth capsule, each of which is different from each other in wall

materials, amounts of wall materials, ratios of wall materials, core modifiers, scavengers, active materials, curing temperatures, heating rates, curing times or a combination thereof.

[0012] Any of the above liquid fabric conditioning compositions can also contain a malodor counteractant. Nonlimiting malodor counteractants are 1-cyclohexylethan-1-yl butyrate, 1-cyclohexylethan-1-yl acetate, 1-cyclohexylethan-1-ol, 1-(4'-methylethyl)cyclohexylethan-1-yl propionate, 2'-hydroxy-1'-ethyl(2-phenoxy)acetate, and any combination thereof.

[0013] A deposition aid can also be included in any of the fabric conditioning compositions. Exemplary deposition aids are anionic, cationic, nonionic and zwitterionic polymers such as polyquaternium-6, polyquaternium-47, polyquaternium-53, polyvinylamine, poly(vinsylamine-co-vinylformamide), and a combination thereof.

[0014] Another aspect of this invention relates to a method of stabilizing the viscosity of a liquid fabric conditioning composition, the method comprising the steps of: (i) providing a liquid fabric conditioning base containing 1-30% of a fabric conditioning agent, and (ii) mixing a stabilizing agent with the liquid fabric conditioning base. The stabilizing agent, being at a concentration of 20 ppm or greater, is a multi-functional amine, mono-functional amine, or an amino acid.

[0015] The details of one or more embodiments of the invention are set forth in the description below. Other features, objects, and advantages of the invention will be apparent from the description and the claims.

DETAILED DESCRIPTION OF THE INVENTION

[0016] It has been found that certain stabilizing agents improve the viscosity stability of liquid fabric conditioning compositions and thus extend the shelf life the conditioning compositions.

[0017] *Stabilizing Agents.* The stabilizing agent in liquid fabric conditioning composition is present in an amount effective to stabilize the viscosity of the composition for an extended period of time (*e.g.*, at least one week, at least four weeks, and at least eight weeks). The viscosity can be deemed stable if it increases during the test period no more than 100 mPas (*e.g.*, no more than 50 mPas and no more than 20 mPas) measured at a constant shear rate of 20 second⁻¹ for 3 minutes at 25°C. The viscosity can also be

deemed stabilized if it increases no more than 50% (*e.g.*, no more than 30% and no more than 20%) as compared to the initial viscosity.

[0018] The effective amount is typically 1 ppm or greater (*e.g.*, 20 ppm or greater, 20 ppm to 20%, 50 ppm to 10%, 50 ppm to 2%, 50 ppm to 1%, 50 to 2000 ppm, and 50 to 1000 ppm). Its concentration in a consumer product can be 20 ppm to 2% (*e.g.*, 50 ppm to 2%, 50 ppm to 1%, 50 to 2000 ppm, and 50 to 1000 ppm).

[0019] Useful stabilizing agents include multi-functional amines, amino acids/peptides, mono-functional amines, polymers, and a polymeric mixture. These stabilizing agents are in presence in the compositions as free compounds, which are not covalently attached to the capsule walls, being part of the capsule walls, or encapsulated in capsules.

[0020] Multi-functional amines are those having at least an amine group (primary, secondary, or tertiary) and one or more other functional groups such as an amine and hydroxyl group. Exemplary multi-functional amines include hexamethylenediamine, hexaethylenediamine, ethylenediamine, 1,3-diaminopropane, 1,4-diamino-butane, diethylenetriamine, pentaethylenehexamine, bis(3-aminopropyl)amine, bis(hexanethylene)triamine, tris(2-aminoethyl)amine, triethylene-tetramine, N,N'-bis(3-aminopropyl)-1,3-propanediamine, tetraethylenepentamine, amino-2-methyl-1-propanol branched polyethylenimine, chitosan, 1,3-diamino-guanidine, 1,1-dimethylbiguanide, and guanidine. Suitable amino acids/peptides include arginine, lysine, histidine, ornithine, nisin, and gelatin. Suitable stabilizing polymers include polyvinylpyrrolidone, polyvinylpyridine-N-oxide, and polyvinyl imidazolium. These polymers sometimes are used in combination with a second polymer (*e.g.*, a block copolymer) such that the second polymer.

[0021] Mono-functional amines have a single amine group. Examples include C1-C20 primary, secondary, or tertiary amines, each of which typically has a molecular weight of 30 to 800 Daltons (*e.g.*, 31 to 500 Daltons and 31 to 300 Daltons). They can be linear, branched, cyclic, acyclic, saturated, unsaturated, aliphatic, and/or aromatic. Nonlimiting examples are methylamine, dimethylamine, trimethylamine, ethylamine, diethylamine, triethylamine, propylamine, isopropylamine, butylamine, dodecylamine, tetradecylamine, aniline, 4-methylaniline, 2-nitroaniline, diphenyl amine, pyrrolidone, piperidine, and morpholine.

[0022] *Fabric Conditioning Agents.* The liquid fabric conditioning compositions of this invention contains at least one fabric conditioning agent, preferably at a concentration of 1 to 30% (e.g., 4 to 20%, 4 to 10%, and 8 to 15%). It would be obvious to a skilled person in the art to determine the concentration of a fabric conditioning agent while keeping its conditioning benefits and also maintaining a reasonable stability and shelf life.

[0023] Suitable fabric conditioning agents include cationic surfactants. Non-limiting examples are quaternary ammonium compounds such as alkylated quaternary ammonium compounds, ring or cyclic quaternary ammonium compounds, aromatic quaternary ammonium compounds, diquaternary ammonium compounds, alkoxyated quaternary ammonium compounds, amidoamine quaternary ammonium compounds, ester quaternary ammonium compounds, and mixtures thereof. Fabric softening compositions, and components thereof, are generally described in US 2004/0204337 and US 2003/0060390. Suitable softening agents include esterquats such as Rewoquat WE 18 commercially available from Evonik Industries and Stepantex SP-90 commercially available from Stepan Company.

[0024] The liquid fabric conditioning compositions can further contain any of the capsules described below.

Core-Shell Encapsulation Systems.

[0025] The capsules can be prepared following encapsulation procedures known in the art, see for example US Patent Nos. 2,800,457, 3,870,542, 3,516,941, 3,415,758, 3,041,288, 5,112,688, 6,329,057, and 6,261,483. Wall forming materials include melamine formaldehyde, polyurethane, polysiloxanes, polyurea, polyamide, polyimide, polyvinyl alcohol, polyanhydride, polyolefin, polysulfone, polysaccharide, protein, polylactide (PLA), polyglycolide (PGA), polyorthoester, polyphosphazene, silicone, lipid, modified cellulose, gums, polystyrene, and polyesters or combinations of these materials. Other polymeric materials that are functional are ethylene maleic anhydride copolymer, styrene maleic anhydride copolymer, ethylene vinyl acetate copolymer, and lactide glycolide copolymer. Biopolymers that are derived from alginate, chitosan, collagen, dextran, gelatin, and starch can also be used as the encapsulating materials. Additionally, capsules can be made via the simple or complex coacervation of gelatin. Preferred encapsulating wall polymers include those formed from isocyanates, acrylates,

acrylamide, acrylate-co-acrylamide, hydrogel monomers, sol-gel precursors, gelatin, melamine-formaldehyde or urea-formaldehyde condensates, as well as similar types of aminoplasts.

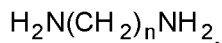
Polyurea/Polyurethane Capsules.

[0026] Polyurea capsules can be prepared using multi-functional isocyanates and multi-functional amines. See WO 2004/054362; EP 0 148149; EP 0 017 409 B1; US Patent Nos. 4,417,916, 4,124,526, 4,285,720, 4,681,806, 5,583,090, 6,340,653 6,566,306, 6,730,635, 8,299,011, WO 90/08468, and WO 92/13450.

[0027] These isocyanates contain two or more isocyanate (-NCO) groups. Suitable isocyanates include, for example, 1,5-naphthylene diisocyanate, 4,4'-diphenylmethane diisocyanate (MDI), hydrogenated MDI (H12MDI), xylylene diisocyanate (XDI), tetramethylxylol diisocyanate (TMXDI), 4,4'-diphenyldimethylmethane diisocyanate, di- and tetraalkyldiphenylmethane diisocyanate, 4,4'-dibenzyl diisocyanate, 1,3-phenylene diisocyanate, 1,4-phenylene diisocyanate, the isomers of tolylene diisocyanate (TDI), optionally in a mixture, 1-methyl-2,4-diisocyanatocyclohexane, 1,6-diisocyanato-2,2,4-trimethylhexane, 1,6-diisocyanato-2,4,4-trimethylhexane, 1-isocyanatomethyl-3-isocyanato-1,5,5-trimethylcyclohexane, chlorinated and brominated diisocyanates, phosphorus-containing diisocyanates, 4,4'-diisocyanatophenylperfluoroethane, tetramethoxybutane 1,4-diisocyanate, butane 1,4-diisocyanate, hexane 1,6-diisocyanate (HDI), dicyclohexylmethane diisocyanate, cyclohexane 1,4-diisocyanate, ethylene diisocyanate, phthalic acid bisisocyanatoethyl ester, also polyisocyanates with reactive halogen atoms, such as 1-chloromethylphenyl 2,4-diisocyanate, 1-bromomethylphenyl 2,6-diisocyanate, and 3,3-bischloromethyl ether 4,4'-diphenyldiisocyanate. Sulfur-containing polyisocyanates are obtained, for example, by reacting hexamethylene diisocyanate with thiodiglycol or dihydroxydihexyl sulfide. Further suitable diisocyanates are trimethylhexamethylene diisocyanate, 1,4-diisocyanatobutane, 1,2-diisocyanatododecane and dimer fatty acid diisocyanate.

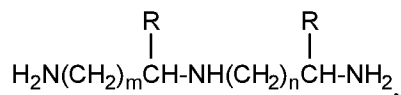
[0028] The multi-functional amines contains two or more amine groups including -NH₂ and -RNH, R being substituted and unsubstituted C₁-C₂₀ alkyl, C₁-C₂₀ heteroalkyl, C₁-C₂₀ cycloalkyl, 3- to 8-membered heterocycloalkyl, aryl, and heteroaryl.

[0029] Water soluble diamines are one class of amines of use in this invention as the amine is usually present in the aqueous phase. One class of such amine is of the type:



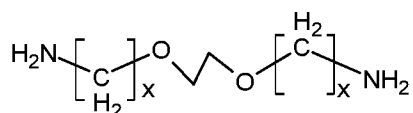
where n is ≥ 1 . When n is 1, the amine is a diamine, ethylene diamine. When n is 2, the amine is diamine propane and so on. Exemplary amines of this type include, but are not limited to, ethylenediamine, 1,3-diaminopropane, 1,4-diaminobutane, hexamethylene diamine, hexamethylene diamine, and pentaethylenehexamine. In particular embodiments of this invention, the preferred n is 6, where the amine is a hexamethylene diamine.

[0030] Amines that have a functionality greater than 2, but less than 3 and which may provide a degree of cross linking in the shell wall are the polyalkylene polyamines of the type:



where R equals hydrogen or $-\text{CH}_3$, m is 1-5 and n is 1-5, *e.g.*, diethylene triamine, triethylene tetraamine and the like. Exemplary amines of this type include, but are not limited to diethylenetriamine, bis(3-aminopropyl)amine, bis(hexamethylene)triamine.

[0031] Another class of amine that can be used in the invention is polyetheramines. They contain primary amino groups attached to the end of a polyether backbone. The polyether backbone is normally based on either propylene oxide (PO), ethylene oxide (EO), or mixed PO/EO. The ether amine can be monoamine, diamine, or triamine, based on this core structure. An example is:



Exemplary polyetheramines include 2,2'-ethylenedioxy)bis (ethylamine) and 4,7,10-trioxa-1,13-tridecanediamine.

[0032] Other suitable amines include, but are not limited to, tris(2-aminoethyl)amine, triethylenetetramine, N,N'-bis(3-aminopropyl)-1,3-propanediamine, tetraethylene pentamine, 1,2-diaminopropane, N,N,N',N'-tetrakis(2-hydroxyethyl)ethylene diamine, N,N,N',N'-tetrakis(2-hydroxypropyl)ethylene diamine, branched polyethylenimine, 2,4-diamino-6-hydroxypyrimidine and 2,4,6-triaminopyrimidine.

[0033] Amphoteric amines, *i.e.*, amines that can react as an acid as well as a base, are another class of amines of use in this invention. Examples of amphoteric amines include

proteins and amino acids such as gelatin, L-lysine, L-arginine, L-lysine monohydrochloride, arginine monohydrochloride and ornithine monohydrochloride.

[0034] Guanidine amines and guanidine salts are yet another class of amines of use in this invention. Exemplary guanidine amines and guanidine salts include, but are not limited to, 1,3-diaminoguanidine monohydrochloride, 1,1-dimethylbiguanide hydrochloride, guanidine carbonate and guanidine hydrochloride.

[0035] Commercially available examples of amines include JEFFAMINE EDR-148 (where $x=2$), JEFFAMINE EDR-176 (where $x=3$) (from Huntsman). Other polyether amines include the JEFFAMINE ED Series, and JEFFAMINE TRIAMINES.

[0036] Alcohols of use as cross-linking agents typically have at least two nucleophilic centers. Exemplary alcohols include, but are not limited to, ethylene glycol, hexylene glycol, pentaerythritol, glucose, sorbitol, and 2-aminoethanol.

[0037] The preparation of polyurethane capsules can be carried out by reacting one or more of the above-referenced isocyanates with a diol or polyol in the presence of a catalyst. Diols or polyols of use in the present invention have a molecular weight in the range of 200-2000. Exemplary diols include, but are not limited to, ethylene glycol, diethylene glycol, propylene glycol, 1,4-butane diol, 1,4 hexane diol, dipropylene glycol, cyclohexyl 1,4 dimethanol, and 1,8 octane diol. Exemplary polyols include, but are not limited to, poly (ethylene glycols), poly (propylene glycols), and poly (tetramethylene glycols).

[0038] Catalysts suitable for use in the invention are amino or organometallic compounds and include, for example, 1,4-diazabicyclo[2.2.2]octane (*e.g.*, DABCO, Air Products, Allentown, PA), N,N-dimethylaminoethanol, N,N-dimethylcyclohexylamine, bis-(2-dimethylaminoethyl) ether, N,N dimethylacetylamine, stannous octoate and dibutyltin dilaurate.

[0039] Table 1 below lists typical cross-liners useful for preparing the polyurea and polyurethane walls.

TABLE 1

Active Hydrogen Compound	Typical Structure	Rel Rxn Rate w/ p-MDI @ 25°C
1° Aliphatic Amine	R-NH ₂	100,000
2° Aliphatic Amine	R ₁ R ₂ NH	20,000-50,000

1° Aromatic Amine	Ar-NH ₂	200-300
1° Hydroxyl	R-CH ₂ -OH	100
Water	H-O-H	100
2° Aromatic Amine	Ar-NH-R or Ar-NH-Ar'	100
Carboxylic Acid	R-COOH	40
2° Hydroxyl	RR'CH-OH	30
Ureas	R-NH-C(O)-NH-R'	15
3° Hydroxyl	R ₃ C-OH	0.5
Urethanes	R-NH-C(O)-O-R'	0.3
Amide	RC(O)-NH ₂	0.1

Aminoplasts and Gelatin

[0040] A representative process used for aminoplast encapsulation is disclosed in US 3,516,941, though it is recognized that many variations with regard to materials and process steps are possible. Another encapsulation process, i.e., gelatin encapsulation, is disclosed in US 2,800,457. Both processes are discussed in the context of fragrance encapsulation for use in consumer products in US Patent Nos. 4,145,184 and 5,112,688 respectively. Polymer systems are well-known in the art and non-limiting examples of these include aminoplast capsules and encapsulated particles as disclosed in GB 2006709 A; the production of micro-capsules having walls comprising styrene-maleic anhydride reacted with melamine-formaldehyde precondensates as disclosed in US 4,396,670; an acrylic acid-acrylamide copolymer, cross-linked with a melamine-formaldehyde resin as disclosed in US 5,089,339; capsules composed of cationic melamine-formaldehyde condensates as disclosed in US 5,401,577; melamine formaldehyde microencapsulation as disclosed in US 3,074,845; amido-aldehyde resin in-situ polymerized capsules disclosed in EP 0 158 449 A1; etherified urea-formaldehyde polymer as disclosed in US 5,204,185; melamine-formaldehyde microcapsules as described in US 4,525,520; cross-linked oil-soluble melamine-formaldehyde precondensate as described in US 5,011,634; capsule wall material formed from a complex of cationic and anionic melamine-formaldehyde precondensates that are then cross-linked as disclosed in US 5,013,473; polymeric shells made from addition polymers such as condensation polymers, phenolic aldehydes, urea aldehydes or acrylic polymer as disclosed in US 3,516,941; urea-

formaldehyde capsules as disclosed in EP 0 443 428 A2; melamine-formaldehyde chemistry as disclosed in GB 2 062 570 A; and capsules composed of polymer or copolymer of styrene sulfonic acid in acid or salt form, and capsules cross-linked with melamine-formaldehyde as disclosed in US 4,001,140.

Urea-formaldehyde and melamine-formaldehyde Capsules

[0041] Urea-formaldehyde and melamine-formaldehyde pre-condensate microcapsule shell wall precursors are prepared by means of reacting urea or melamine with formaldehyde where the mole ratio of melamine or urea to formaldehyde is in the range of from 10:1 to 1:6, preferably from 1:2 to 1:5. For purposes of practicing this invention, the resulting material has a molecular weight in the range of from 156 to 3000. The resulting material may be used 'as-is' as a cross-linking agent for the aforementioned substituted or un-substituted acrylic acid polymer or copolymer or it may be further reacted with a C₁-C₆ alkanol, *e.g.*, methanol, ethanol, 2-propanol, 3-propanol, 1-butanol, 1-pentanol or 1-hexanol, thereby forming a partial ether where the mole ratio of melamine/urea:formaldehyde:alkanol is in the range of 1:(0.1-6):(0.1-6). The resulting ether moiety-containing product may be used 'as-is' as a cross-linking agent for the aforementioned substituted or un-substituted acrylic acid polymer or copolymer, or it may be self-condensed to form dimers, trimers and/or tetramers which may also be used as cross-linking agents for the aforementioned substituted or un-substituted acrylic acid polymers or co-polymers. Methods for formation of such melamine-formaldehyde and urea-formaldehyde pre-condensates are set forth in US Patent Nos. 3,516,846 and 6,261,483, and Lee et al. (2002) *J. Microencapsulation* 19, 559-569.

[0042] Examples of urea-formaldehyde pre-condensates useful in the practice of this invention are URAC 180 and URAC 186, trademarks of Cytec Technology Corp. of Wilmington, DE. Examples of melamine-formaldehyde pre-condensates useful in the practice of this invention, include, but are not limited to, CYMEL U-60, CYMEL U-64 and CYMEL U-65, trademarks of Cytec Technology Corp. of Wilmington, DE. It is preferable to use, as the precondensate for cross-linking, the substituted or un-substituted acrylic acid polymer or co-polymer. In practicing this invention, the range of mole ratios of urea-formaldehyde precondensate/melamine-formaldehyde pre-condensate to substituted/un-substituted acrylic acid polymer/co-polymer is in the range of from 9:1 to 1:9, preferably from 5:1 to 1:5 and even more preferably from 2:1 to 1:2.

[0043] In one embodiment of the invention, microcapsules with polymer(s) composed of primary and/or secondary amine reactive groups or mixtures thereof and cross-linkers can also be used. See US 2006/0248665. The amine polymers can possess primary and/or secondary amine functionalities and can be of either natural or synthetic origin. Amine-containing polymers of natural origin are typically proteins such as gelatin and albumen, as well as some polysaccharides. Synthetic amine polymers include various degrees of hydrolyzed polyvinyl formamides, polyvinylamines, polyallyl amines and other synthetic polymers with primary and secondary amine pendants. Examples of suitable amine polymers are the LUPAMIN series of polyvinyl formamides available from BASF. The molecular weights of these materials can range from 10,000 to 1,000,000.

[0044] Urea-formaldehyde or melamine-formaldehyde capsules can also include formaldehyde scavengers, which are capable of binding free formaldehyde. When the capsules are for use in aqueous media, formaldehyde scavengers such as sodium sulfite, melamine, glycine, and carboglycidine are suitable. When the capsules are aimed to be used in products having low pH, *e.g.*, fabric care conditioners, formaldehyde scavengers are preferably selected from beta diketones, such as beta-ketoesters, or from 1,3-diols, such as propylene glycol. Preferred beta-ketoesters include alkyl-malonates, alkyl acetoacetates and polyvinyl alcohol acetoacetates.

[0045] As indicated, the capsules of this invention can be prepared by conventional methods to encapsulate fragrances. In some embodiments, the fragrance is encapsulated by a polymer in the presence of a capsule formation aid, *e.g.*, a surfactant or dispersant. Classes of protective colloid or emulsifier of use as surfactants or dispersants include maleic-vinyl copolymers such as the copolymers of vinyl ethers with maleic anhydride or acid, sodium lignosulfonates, maleic anhydride/styrene copolymers, ethylene/maleic anhydride copolymers, and copolymers of propylene oxide, ethylenediamine and ethylene oxide, polyvinylpyrrolidone, polyvinyl alcohols, carboxymethyl cellulose, fatty acid esters of polyoxyethylenated sorbitol and sodium dodecylsulfate.

[0046] Commercially available surfactants include, but are not limited to, sulfonated naphthalene-formaldehyde condensates such as MORWET D425 (Akzo Nobel); partially hydrolyzed polyvinyl alcohols such as MOWIOLs, *e.g.*, MOWIOL 3-83 (Air Products); ethylene oxide-propylene oxide block copolymers or poloxamers such as PLURONIC,

SYNPERONIC or PLURACARE materials (BASF); sulfonated polystyrenes such as FLEXAN II (Akzo Nobel); and ethylene-maleic anhydride polymers such as ZEMAC (Vertellus Specialties Inc.)

[0047] Typically, hydrocolloids or adjuvants are used to improve the colloidal stability of the capsule suspension or slurry against coagulation, sedimentation and creaming. As such, such processing aids can also be used in conjunction with the microcapsules of this invention. As used herein, the term “hydrocolloid” refers to a broad class of water-soluble or water-dispersible polymers having anionic, cationic, zwitterionic or nonionic character. In particular embodiments, the capsule suspension includes a nonionic polymer, cationic polymer, anionic polymer, anionic surfactant, or a combination thereof. In certain embodiments, the nonionic polymer is a polyvinylpyrrolidone (PVP), polyvinyl alcohol (PVA) polyethylene glycol (PEG), Polyethylene oxide (PEO), or polyethylene oxide-polypropylene oxide (PEO-PPO), polyethylene oxide-polypropylene oxide-polyethylene oxide (PEO-PPO-PEO). In other embodiments, the cationic polymer is Polyquaternium-6 (polydiallyldimethylammonium chloride), Polyquaternium-11 (vinyl pyrrolidone/dimethylaminoethyl methacrylate copolymer) or Polyquaternium-47 (acrylic acid/methacrylamidopropyl trimethyl ammonium chloride/methyl acrylate terpolymer). In yet other embodiments, the anionic polymer is a polystyrene sulfonic acid, polyacrylic acid, hyaluronic acid, sodium alginate, or sodium carboxymethylcellulose (CMC). In still other embodiments, the anionic surfactant is sodium laureth sulfate (SLS) or a complex ester of phosphoric acid and ethoxylated cosmetic grade oleyl alcohol (*e.g.*, CRODAFOS 010A-SS-(RB)).

[0048] Other hydrocolloids useful in the present invention include polycarbohydrates, such as starch, modified starch, dextrin, maltodextrin, and cellulose derivatives, and their quaternized forms; natural gums such as alginate esters, carrageenan, xanthanes, agar-agar, pectins, pectic acid, and natural gums such as gum arabic, gum tragacanth and gum karaya, guar gums and quaternized guar gums; gelatin, protein hydrolysates and their quaternized forms; synthetic polymers and copolymers, such as poly(vinyl pyrrolidone-co-vinyl acetate), poly(vinyl alcohol-co-vinyl acetate), poly((met)acrylic acid), poly(maleic acid), poly(alkyl(meth)acrylate-co-(meth)acrylic acid), poly(acrylic acid-co-maleic acid)copolymer, poly(alkyleneoxide), poly(vinylmethylether), poly(vinylether-co-maleic anhydride), and the like, as well as

poly-(ethyleneimine), poly((meth)acrylamide), poly(alkyleneoxide-co-dimethylsiloxane), poly(amino dimethylsiloxane), and their quaternized forms.

[0049] The capsule formation aid may also be used in combination with carboxymethyl cellulose and/or a surfactant during processing to facilitate capsule formation. Examples of surfactants that can be used in combination with the capsule formation aid include, but are not limited to, cetyl trimethyl ammonium chloride (CTAC), poloxamers such as PLURONICS (*e.g.*, PLURONIC F127), PLURAFAC (*e.g.*, PLURAFAC F127), or MIRANET-N, saponins such as QNATURALE (National Starch Food Innovation); or a gum Arabic such as Seyal or Senegal. The amount of surfactant present in the capsule slurry can vary depending on the surfactant used. In some embodiments the amount of surfactant is in the range of 0.05 to 0.2 weight percent, in particular when CTAC is employed. In another embodiment, the amount of surfactant is in the range of 1 to 3 weight percent when a saponin or gum arabic is used.

[0050] When combined with carboxymethyl cellulose (also referred to as CMC), the lighter color polyvinyl alcohol, carboxymethyl cellulose is preferred. In certain embodiments, the carboxymethyl cellulose polymer has a molecular weight range between 90,000 Daltons to 1,500,000 Daltons, more preferably between 250,000 Daltons to 750,000 Daltons and even more preferably between 400,000 Daltons to 750,000 Daltons. The carboxymethyl cellulose polymer has a degree of substitution between 0.1 to 3, more preferably between 0.65 to 1.4, and even more preferably between 0.8 to 1.0.

[0051] The carboxymethyl cellulose polymer is present in the capsule slurry at a level from 0.1 weight percent to 2 weight percent and more preferably from 0.3 weight percent to 0.7 weight percent.

[0052] In some embodiments, CMC-modified microcapsules may provide a perceived fragrance intensity increase of greater than 15%, and more preferably an increase of greater than 25% as compared to microcapsules not including CMC.

[0053] The diameter of the capsules produced in accordance with this invention can vary from 10 nanometers to 1000 microns, preferably from 50 nanometers to 150 microns and is even more preferably from 2 to 15 microns. The capsule distribution can be narrow, broad, or multi-modal. In particular embodiments, the delivery system of the invention has a multi-modal distribution indicative of different types of capsule chemistries.

[0054] In some embodiments, the capsule suspension prepared in accordance with the present invention is subsequently purified. Purification can be achieved by washing the capsule slurry with water, *e.g.*, deionized or double deionized water, until a neutral pH is achieved. For the purposes of the present invention, the capsule suspension can be washed using any conventional method including the use of a separatory funnel, filter paper, centrifugation and the like. The capsule suspension can be washed one, two, three, four, five, six, seven, eight, nine, ten or more times until a neutral pH, *i.e.*, $\text{pH } 7 \pm 0.5$, is achieved. The pH of the purified capsules can be determined using any conventional method including, but not limited to pH paper, pH indicators, or a pH meter.

[0055] A capsule suspension of this invention is “purified” in that it is 80%, 90%, 95%, 97%, 98% or 99% homogeneous to capsules. In accordance with the present invention, purity is achieved by washing the capsules until a neutral pH is achieved, which is indicative of removal of unwanted impurities and/or starting materials, *e.g.*, polyisocyanate, cross-linking agent and the like.

[0056] In certain embodiments of this invention, the purification of the capsules includes the additional step of adding a salt to the capsule suspension prior to the step of washing the capsule suspension with water. Exemplary salts of use in this step of the invention include, but are not limited to, sodium chloride, potassium chloride or bi-sulphite salts.

Active materials.

[0057] Active materials include, but are not limited to, fragrances, malodor counteractants and any combination thereof. Suitable fragrances include without limitation, any combination of fragrance oil, essential oil, plant extract or mixture thereof that is compatible with, and capable of being encapsulated by a polymer. Individual perfume ingredients that can be included in the capsules of this invention include fragrances containing:

[0058] i) hydrocarbons, such as, for example, 3-carene, α -pinene, β -pinene, α -terpinene, γ -terpinene, p-cymene, bisabolene, camphene, caryophyllene, cedrene, farnesene, limonene, longifolene, myrcene, ocimene, valencene, (E,Z)-1,3,5-undecatriene, styrene, and diphenylmethane;

[0059] ii) aliphatic alcohols, such as, for example, hexanol, octanol, 3-octanol, 2,6-dimethylheptanol, 2-methyl-2-heptanol, 2-methyl-2-octanol, (E)-2-hexenol, (E)- and (Z)-

3-hexenol, 1-octen-3-ol, a mixture of 3,4,5,6,6-pentamethyl-3/4-hepten-2-ol and 3,5,6,6-tetramethyl-4-methyleneheptan-2-ol, (E,Z)-2,6-nonadienol, 3,7-dimethyl-7-methoxyoctan-2-ol, 9-decenol, 10-undecenol, 4-methyl-3-decen-5-ol, aliphatic aldehydes and their acetals such as for example hexanal, heptanal, octanal, nonanal, decanal, undecanal, dodecanal, tridecanal, 2-methyloctanal, 2-methylnonanal, (E)-2-hexenal, (Z)-4-heptenal, 2,6-dimethyl-5-heptenal, 10-undecenal, (E)-4-decenal, 2-dodecenal, 2,6,10-trimethyl-5,9-undecadienal, heptanal-diethylacetal, 1,1-dimethoxy-2,2,5-trimethyl-4-hexene, and citronellyl oxyacetaldehyde;

[0060] iii) aliphatic ketones and oximes thereof, such as, for example, 2-heptanone, 2-octanone, 3-octanone, 2-nonanone, 5-methyl-3-heptanone, 5-methyl-3-heptanone oxime, 2,4,4,7-tetramethyl-6-octen-3-one, aliphatic sulfur-containing compounds, such as for example 3-methylthiohexanol, 3-methylthiohexyl acetate, 3-mercaptohexanol, 3-mercaptohexyl acetate, 3-mercaptohexyl butyrate, 3-acetylthiohexyl acetate, 1-menthene-8-thiol, and aliphatic nitriles (*e.g.*, 2-nonenenitrile, 2-tridecenenitrile, 2,12-tridecenenitrile, 3,7-dimethyl-2,6-octadienenitrile, and 3,7-dimethyl-6-octenenitrile);

[0061] iv) aliphatic carboxylic acids and esters thereof, such as, for example, (E)- and (Z)-3-hexenylformate, ethyl acetoacetate, isoamyl acetate, hexyl acetate, 3,5,5-trimethylhexyl acetate, 3-methyl-2-butenyl acetate, (E)-2-hexenyl acetate, (E)- and (Z)-3-hexenyl acetate, octyl acetate, 3-octyl acetate, 1-octen-3-yl acetate, ethyl butyrate, butyl butyrate, isoamyl butyrate, hexylbutyrate, (E)- and (Z)-3-hexenyl isobutyrate, hexyl crotonate, ethylisovalerate, ethyl-2-methyl pentanoate, ethyl hexanoate, allyl hexanoate, ethyl heptanoate, allyl heptanoate, ethyl octanoate, ethyl-(E,Z)-2,4-decadienoate, methyl-2-octinate, methyl-2-noninate, allyl-2-isoamyl oxyacetate, and methyl-3,7-dimethyl-2,6-octadienoate;

[0062] v) acyclic terpene alcohols, such as, for example, citronellol; geraniol; nerol; linalool; lavandulol; nerolidol; farnesol; tetrahydrolinalool; tetrahydrogeraniol; 2,6-dimethyl-7-octen-2-ol; 2,6-dimethyloctan-2-ol; 2-methyl-6-methylene-7-octen-2-ol; 2,6-dimethyl-5,7-octadien-2-ol; 2,6-dimethyl-3,5-octadien-2-ol; 3,7-dimethyl-4,6-octadien-3-ol; 3,7-dimethyl-1,5,7-octatrien-3-ol 2,6-dimethyl-2,5,7-octatrien-1-ol; as well as formates, acetates, propionates, isobutyrate, butyrate, isovalerate, pentanoates, hexanoates, crotonates, tiglinates and 3-methyl-2-butenates thereof;

[0063] vi) acyclic terpene aldehydes and ketones, such as, for example, geranial, neral, citronellal, 7-hydroxy-3,7-dimethyloctanal, 7-methoxy-3,7-dimethyloctanal, 2,6,10-trimethyl-9-undecenal, α -sinensal, β -sinensal, geranylacetone, as well as the dimethyl- and diethylacetals of geranial, neral and 7-hydroxy-3,7-dimethyloctanal;

[0064] vii) cyclic terpene alcohols, such as, for example, menthol, isopulegol, α -terpineol, terpinen-4-ol, menthan-8-ol, menthan-1-ol, menthan-7-ol, borneol, isoborneol, linalool oxide, nopol, cedrol, ambrinol, vetiverol, guaiol, and the formates, acetates, propionates, isobutyrate, butyrate, isovalerate, pentanoate, hexanoate, crotonate, tiglinates and 3-methyl-2-butenates of α -terpineol, terpinen-4-ol, menthan-8-ol, menthan-1-ol, menthan-7-ol, borneol, isoborneol, linalool oxide, nopol, cedrol, ambrinol, vetiverol, and guaiol;

[0065] viii) cyclic terpene aldehydes and ketones, such as, for example, menthone, isomenthone, 8-mercaptomenthan-3-one, carvone, camphor, fenchone, α -ionone, β -ionone, α -n-methylionone, β -n-methylionone, α -isomethylionone, β -isomethylionone, α -irone, α -damoscone, β -damoscone, β -damoscenone, δ -damoscone, γ -damoscone, 1-(2,4,4-trimethyl-2-cyclohexen-1-yl)-2-buten-1-one, 1,3,4,6,7,8a-hexahydro-1,1,5,5-tetramethyl-2H-2,4a-methanonaphthalen-8(5H)-one, nootkatone, dihydronootkatone; acetylated cedarwood oil (cedryl methyl ketone);

[0066] ix) cyclic alcohols, such as, for example, 4-tert-butylcyclohexanol, 3,3,5-trimethylcyclohexanol, 3-isocamphylcyclohexanol, 2,6,9-trimethyl-Z2,Z5,E9-cyclododecatrien-1-ol, 2-isobutyl-4-methyltetrahydro-2H-pyran-4-ol;

[0067] x) cycloaliphatic alcohols, such as, for example, α , 3,3-trimethylcyclohexylmethanol, 2-methyl-4-(2,2,3-trimethyl-3-cyclopent-1-yl)butanol, 2-methyl-4-(2,2,3-trimethyl-3-cyclopent-1-yl)-2-buten-1-ol, 2-ethyl-4-(2,2,3-trimethyl-3-cyclopent-1-yl)-2-buten-1-ol, 3-methyl-5-(2,2,3-trimethyl-3-cyclopent-1-yl)pentan-2-ol, 3-methyl-5-(2,2,3-trimethyl-3-cyclopent-1-yl)-4-penten-2-ol, 3,3-dimethyl-5-(2,2,3-trimethyl-3-cyclopent-1-yl)-4-penten-2-ol, 1-(2,2,6-trimethylcyclohexyl)pentan-3-ol, 1-(2,2,6-trimethylcyclohexyl)hexan-3-ol;

[0068] xi) cyclic and cycloaliphatic ethers, such as, for example, cineole, cedryl methyl ether, cyclododecyl methyl ether;

[0069] xii) (ethoxymethoxy)cyclododecane; α -cedrene epoxide, 3a,6,6,9a-tetramethyldodecahydronaphtho[2,1-b]furan, 3a-ethyl-6,6,9a-

trimethyldodecahydronaphtho[2,1-b]furan, 1,5,9-trimethyl-13-oxabicyclo[10.1.0]-trideca-4,8-diene, rose oxide, 2-(2,4-dimethyl-3-cyclohexen-1-yl)-5-methyl-5-(1-methylpropyl)-1,3-dioxan;

[0070] xiii) cyclic ketones, such as, for example, 4-tert-butylcyclohexanone, 2,2,5-trimethyl-5-pentylcyclopentanone, 2-heptylcyclopentanone, 2-pentylcyclopentanone, 2-hydroxy-3-methyl-2-cyclopenten-1-one, 3-methyl-cis-2-penten-1-yl-2-cyclopenten-1-one, 3-methyl-2-pentyl-2-cyclopenten-1-one, 3-methyl-4-cyclopentadecenone, 3-methyl-5-cyclopentadecenone, 3-methylcyclopentadecanone, 4-(1-ethoxyvinyl)-3,3,5,5-tetramethylcyclohexanone, 4-tert-pentylcyclohexanone, 5-cyclohexadecen-1-one, 6,7-dihydro-1,1,2,3,3-pentamethyl-4(5H)-indanone, 5-cyclohexadecen-1-one, 8-cyclohexadecen-1-one, 9-cycloheptadecen-1-one, cyclopentadecanone, cycloaliphatic aldehydes, such as, for example, 2,4-dimethyl-3-cyclohexene carbaldehyde, 2-methyl-4-(2,2,6-trimethyl-cyclohexen-1-yl)-2-butenal, 4-(4-hydroxy-4-methylpentyl)-3-cyclohexene carbaldehyde, 4-(4-methyl-3-penten-1-yl)-3-cyclohexene carbaldehyde;

[0071] xiv) cycloaliphatic ketones, such as, for example, 1-(3,3-dimethylcyclohexyl)-4-penten-1-one, 1-(5,5-dimethyl-1-cyclohexen-1-yl)-4-penten-1-one, 2,3,8,8-tetramethyl-1,2,3,4,5,6,7,8-octahydro-2-naphthalenyl methyl-ketone, methyl-2,6,10-trimethyl-2,5,9-cyclododecatrienyl ketone, tert-butyl-(2,4-dimethyl-3-cyclohexen-1-yl)ketone;

[0072] xv) esters of cyclic alcohols, such as, for example, 2-tert-butylcyclohexyl acetate, 4-tert-butylcyclohexyl acetate, 2-tert-pentylcyclohexyl acetate, 4-tert-pentylcyclohexyl acetate, decahydro-2-naphthyl acetate, 3-pentyltetrahydro-2H-pyran-4-yl acetate, decahydro-2,5,5,8a-tetramethyl-2-naphthyl acetate, 4,7-methano-3a,4,5,6,7,7a-hexahydro-5 or 6-indenyl acetate, 4,7-methano-3a,4,5,6,7,7a-hexahydro-5 or 6-indenyl propionate, 4,7-methano-3a,4,5,6,7,7a-hexahydro-5 or 6-indenyl-isobutyrate, 4,7-methanooctahydro-5 or 6-indenyl acetate;

[0073] xvi) esters of cycloaliphatic carboxylic acids, such as, for example, allyl 3-cyclohexyl-propionate, allyl cyclohexyl oxyacetate, methyl dihydrojasmonate, methyl jasmonate, methyl 2-hexyl-3-oxocyclopentanecarboxylate, ethyl 2-ethyl-6,6-dimethyl-2-cyclohexenecarboxylate, ethyl 2,3,6,6-tetramethyl-2-cyclohexenecarboxylate, ethyl 2-methyl-1,3-dioxolane-2-acetate;

[0074] xvii) aromatic and aliphatic alcohols, such as, for example, benzyl alcohol, 1-phenylethyl alcohol, 2-phenylethyl alcohol, 3-phenylpropanol, 2-phenylpropanol, 2-

phenoxyethanol, 2,2-dimethyl-3-phenylpropanol, 2,2-dimethyl-3-(3-methylphenyl)propanol, 1,1-dimethyl-2-phenylethyl alcohol, 1,1-dimethyl-3-phenylpropanol, 1-ethyl-1-methyl-3-phenylpropanol, 2-methyl-5-phenylpentanol, 3-methyl-5-phenylpentanol, 3-phenyl-2-propen-1-ol, 4-methoxybenzyl alcohol, 1-(4-isopropylphenyl)ethanol;

[0075] xviii) esters of aliphatic alcohols and aliphatic carboxylic acids, such as, for example, benzyl acetate, benzyl propionate, benzyl isobutyrate, benzyl isovalerate, 2-phenylethyl acetate, 2-phenylethyl propionate, 2-phenylethyl isobutyrate, 2-phenylethyl isovalerate, 1-phenylethyl acetate, α -trichloromethylbenzyl acetate, α,α -dimethylphenylethyl acetate, α,α -dimethylphenylethyl butyrate, cinnamyl acetate, 2-phenoxyethyl isobutyrate, 4-methoxybenzyl acetate, araliphatic ethers, such as for example 2-phenylethyl methyl ether, 2-phenylethyl isoamyl ether, 2-phenylethyl-1-ethoxyethyl ether, phenylacetaldehyde dimethyl acetal, phenylacetaldehyde diethyl acetal, hydratropaaldehyde dimethyl acetal, phenylacetaldehyde glycerol acetal, 2,4,6-trimethyl-4-phenyl-1,3-dioxane, 4,4a,5,9b-tetrahydroindeno[1,2-d]-m-dioxin, 4,4a,5,9b-tetrahydro-2,4-dimethylindeno[1,2-d]-m-dioxin;

[0076] xix) aromatic and aliphatic aldehydes, such as, for example, benzaldehyde; phenylacetaldehyde, 3-phenylpropanal, hydratropaldehyde, 4-methylbenzaldehyde, 4-methylphenylacetaldehyde, 3-(4-ethylphenyl)-2,2-dimethylpropanal, 2-methyl-3-(4-isopropylphenyl)propanal, 2-methyl-3-(4-tert-butylphenyl)propanal, 3-(4-tert-butylphenyl)propanal, cinnamaldehyde, α -butylcinnamaldehyde, α -amylcinnamaldehyde, α -hexylcinnamaldehyde, 3-methyl-5-phenylpentanal, 4-methoxybenzaldehyde, 4-hydroxy-3-methoxybenzaldehyde, 4-hydroxy-3-ethoxybenzaldehyde, 3,4-methylene-dioxybenzaldehyde, 3,4-dimethoxybenzaldehyde, 2-methyl-3-(4-methoxyphenyl)propanal, 2-methyl-3-(4-methylendioxyphenyl)propanal;

[0077] xx) aromatic and aliphatic ketones, such as, for example, acetophenone, 4-methylacetophenone, 4-methoxyacetophenone, 4-tert-butyl-2,6-dimethylacetophenone, 4-phenyl-2-butanone, 4-(4-hydroxyphenyl)-2-butanone, 1-(2-naphthalenyl)ethanone, benzophenone, 1,1,2,3,3,6-hexamethyl-5-indanyl methyl ketone, 6-tert-butyl-1,1-dimethyl-4-indanyl methyl ketone, 1-[2,3-dihydro-1,1,2,6-tetramethyl-3-(1-methylethyl)-1H-5-indenyl]ethanone, 5',6',7',8'-tetrahydro-3',5',5',6',8',8'-hexamethyl-2-acetonaphthone;

[0078] xxi) aromatic and aliphatic carboxylic acids and esters thereof, such as, for example, benzoic acid, phenylacetic acid, methyl benzoate, ethyl benzoate, hexyl benzoate, benzyl benzoate, methyl phenylacetate, ethyl phenylacetate, geranyl phenylacetate, phenylethyl phenylacetate, methyl cinnamate, ethyl cinnamate, benzyl cinnamate, phenylethyl cinnamate, cinnamyl cinnamate, allyl phenoxyacetate, methyl salicylate, isoamyl salicylate, hexyl salicylate, cyclohexyl salicylate, cis-3-hexenyl salicylate, benzyl salicylate, phenylethyl salicylate, methyl 2,4-dihydroxy-3,6-dimethylbenzoate, ethyl 3-phenylglycidate, ethyl 3-methyl-3-phenylglycidate;

[0079] xxii) nitrogen-containing aromatic compounds, such as, for example, 2,4,6-trinitro-1,3-dimethyl-5-tert-butylbenzene, 3,5-dinitro-2,6-dimethyl-4-tert-butylacetophenone, cinnamionitrile, 5-phenyl-3-methyl-2-pentenitrile, 5-phenyl-3-methylpentanonitrile, methyl anthranilate, methyl-N-methylantranilate, Schiff's bases of methyl anthranilate with 7-hydroxy-3,7-dimethyloctanal, 2-methyl-3-(4-tert-butylphenyl)propanal or 2,4-dimethyl-3-cyclohexene carbaldehyde, 6-isopropylquinoline, 6-isobutylquinoline, 6-sec-butylquinoline, indole, skatole, 2-methoxy-3-isopropylpyrazine, 2-isobutyl-3-methoxypyrazine;

[0080] xxiii) phenols, phenyl ethers and phenyl esters, such as, for example, estragole, anethole, eugenol, eugenyl methyl ether, isoeugenol, isoeugenol methyl ether, thymol, carvacrol, diphenyl ether, beta-naphthyl methyl ether, beta-naphthyl ethyl ether, beta-naphthyl isobutyl ether, 1,4-dimethoxybenzene, eugenyl acetate, 2-methoxy-4-methylphenol, 2-ethoxy-5-(1-propenyl)phenol, p-cresyl phenylacetate;

[0081] xxiv) heterocyclic compounds, such as, for example, 2,5-dimethyl-4-hydroxy-2H-furan-3-one, 2-ethyl-4-hydroxy-5-methyl-2H-furan-3-one, 3-hydroxy-2-methyl-4H-pyran-4-one, 2-ethyl-3-hydroxy-4H-pyran-4-one;

[0082] xxv) lactones, such as, for example, 1,4-octanolide, 3-methyl-1,4-octanolide, 1,4-nonanolide, 1,4-decanolide, 8-decen-1,4-olide, 1,4-undecanolide, 1,4-dodecanolide, 1,5-decanolide, 1,5-dodecanolide, 1,15-pentadecanolide, cis- and trans-11-pentadecen-1,15-olide, cis- and trans-12-pentadecen-1,15-olide, 1,16-hexadecanolide, 9-hexadecen-1,16-olide, 10-oxa-1,16-hexadecanolide, 11-oxa-1,16-hexadecanolide, 12-oxa-1,16-hexadecanolide, ethylene-1,12-dodecanedioate, ethylene-1,13-tridecanedioate, coumarin, 2,3-dihydrocoumarin, and octahydrocoumarin; and

[0083] xxvi) essential oils, concretes, absolutes, resins, resinoids, balsams, tinctures such as for example ambergris tincture, amyris oil, angelica seed oil, angelica root oil, aniseed oil, valerian oil, basil oil, tree moss absolute, bay oil, armoise oil, benzoe resinoid, bergamot oil, beeswax absolute, birch tar oil, bitter almond oil, savory oil, buchu leaf oil, cabreuva oil, cade oil, calamus oil, camphor oil, cananga oil, cardamom oil, cascarilla oil, cassia oil, cassie absolute, castoreum absolute, cedar leaf oil, cedar wood oil, cistus oil, citronella oil, lemon oil, copaiba balsam, copaiba balsam oil, coriander oil, costus root oil, cumin oil, cypress oil, davana oil, dill weed oil, dill seed oil, eau de brouts absolute, oakmoss absolute, elemi oil, estragon oil, eucalyptus citriodora oil, eucalyptus oil (cineole type), fennel oil, fir needle oil, galbanum oil, galbanum resin, geranium oil, grapefruit oil, guaiacwood oil, gurjun balsam, gurjun balsam oil, helichrysum absolute, helichrysum oil, ginger oil, iris root absolute, iris root oil, jasmine absolute, calamus oil, blue camomile oil, Roman camomile oil, carrot seed oil, cascarilla oil, pine needle oil, spearmint oil, caraway oil, labdanum oil, labdanum absolute, labdanum resin, lavandin absolute, lavandin oil, lavender absolute, lavender oil, lemon-grass oil, lovage oil, lime oil distilled, lime oil expressed, linaloe oil, Litsea cubeba oil, laurel leaf oil, mace oil, marjoram oil, mandarin oil, massoi (bark) oil, mimosa absolute, ambrette seed oil, musk tincture, clary sage oil, nutmeg oil, myrrh absolute, myrrh oil, myrtle oil, clove leaf oil, clove bud oil, neroli oil, olibanum absolute, olibanum oil, opopanax oil, orange flower absolute, orange oil, origanum oil, palmarosa oil, patchouli oil, perilla oil, Peru balsam oil, parsley leaf oil, parsley seed oil, petitgrain oil, peppermint oil, pepper oil, pimento oil, pine oil, pennyroyal oil, rose absolute, rosewood oil, rose oil, rosemary oil, Dalmatian sage oil, Spanish sage oil, sandal-wood oil, celery seed oil: spike-lavender oil, star anise oil, storax oil, tagetes oil, fir needle oil, tea tree oil, turpentine oil, thyme oil, Tolu balsam, tonka bean absolute, tuberose absolute, vanilla extract, violet leaf absolute, verbena oil, vetiver oil, juniperberry oil, wine lees oil, wormwood oil, wintergreen oil, ylang-ylang oil, hyssop oil, civet absolute, cinnamon leaf oil, cinnamon bark oil, and fractions thereof or ingredients isolated therefrom.

[0084] In some embodiments, the amount of encapsulated fragrance or malodor counteractant is from 0.5% to 80% of the total capsule suspension or capsule slurry, preferably from 5% to 60% of the total capsule suspension or capsule slurry, and even more preferably from 20% to 50% of the total capsule suspension or capsule slurry. In

some embodiments, each encapsulated fragrance or malodor counteractant is an equal proportion of the total encapsulated fragrance oil. By way of illustration, in encapsulation system containing a first and second fragrances, each of the first and second fragrances is 50% of the total encapsulated fragrances. Likewise, in encapsulation system containing a first, second and third fragrances, each of the first, second and third fragrances is ~33% by weight of the total encapsulated fragrances. In other embodiments of this invention, the amount of fragrances in the capsule can be tailored to provide benefits to consumers at various moments. By way of illustration, the first fragrance is 20 to 80% by weight of the total encapsulated fragrances and the second fragrance is 80 to 20%. The ratio between the active material and the fabric conditioning agent can be 1 : 500 to 1 : 2 (e.g., 1 : 250 to 1 : 4 and 1 : 100 to 1 : 8). As an illustration, when the fabric conditioning agent is 5% by weight of the fabric softener, the active material is 0.01 to 2.5%, preferably 0.02 to 1.25% and more preferably 0.1 to 0.63%. As another example, when the fabric conditioning agent is 20% by weight of the fabric softener, the active material is 0.04 to 10%, preferably 0.08 to 5% and more preferably 0.4 to 2.5%. The active material is a fragrance, malodor counteractant or mixture thereof. The liquid fabric softener can have 0.15 to 15% of capsules (e.g., 0.5 to 10%, 0.7 to 5%, and 1 to 3%). When including capsules at these levels, the neat oil equivalent (NOE) in the softener is 0.05 to 5% (e.g., 0.15 to 3.2%, 0.25 to 2%, and 0.3 to 1%).

[0085] In addition to the fragrances and malodor counteractants, solvent materials can also be incorporated into one or more of the capsules. The solvent materials are hydrophobic materials that are miscible with the fragrances or malodor counteractants. The solvent materials serve to increase the compatibility of various active materials, increase the overall hydrophobicity of the mixture containing the active materials, influence the vapor pressure, or serve to structure the mixture. Suitable solvents are those having reasonable affinity for the active materials and a ClogP greater than 2.5, preferably greater than 3.5 and more preferably greater than 5.5. In some embodiments, the solvent is combined with the active materials that have ClogP values as set forth above. It should be noted that selecting a solvent and active material with high affinity for each other will result in improvement in stability. Suitable solvents include triglyceride oil, mono and diglycerides, mineral oil, silicone oil, diethyl phthalate, polyalpha olefins, castor oil, isopropyl myristate, mono-, di- and tri-esters and mixtures

thereof, fatty acids, and glycerine. The fatty acid chain can range from C₄-C₂₆ and can have any level of unsaturation. For instance, one of the following solvents can be used: capric/caprylic triglyceride known as NEOBEE M5 (Stepan Corporation); the CAPMUL series by Abitec Corporation (e.g., CAPMUL MCM); isopropyl myristate; fatty acid esters of polyglycerol oligomers, *e.g.*, R₂CO-[OCH₂-CH(OCOR₁)-CH₂O]_n, where R₁ and R₂ can be H or C₄-C₂₆ aliphatic chains, or mixtures thereof, and n ranges between 2 and 50, preferably 2 and 30; nonionic fatty alcohol alkoxylates like the NEODOL surfactants by BASF; the dobanol surfactants by Shell Corporation or the BIO-SOFT surfactants by Stepan, wherein the alkoxy group is ethoxy, propoxy, butoxy, or mixtures thereof and said surfactants can be end-capped with methyl groups in order to increase their hydrophobicity; di- and tri-fatty acid chain containing nonionic, anionic and cationic surfactants, and mixtures thereof; fatty acid esters of polyethylene glycol, polypropylene glycol, and polybutylene glycol, or mixtures thereof; polyalphaolefins such as the EXXONMOBIL PURESYS PAO line; esters such as the EXXONMOBIL PURESYN esters; mineral oil; silicone oils such polydimethyl siloxane and polydimethylcyclsiloxane; diethyl phthalate; di-octyl adipate and di-isodecyl adipate.

[0086] While no solvent is needed in the core, it is preferable that the level of solvent in the core of the microcapsule product is 80 wt% or less, preferably 50 wt% or less (e.g., 0-20 wt%).

[0087] When the active material is a fragrance, it is preferred that fragrance ingredients within the fragrance having a high ClogP are employed. For instance, the ingredients having a ClogP value between 0.5 to 8 (e.g., between 1 to 12, between 1.5 to 8, between 2 and 7, between 1 and 6, between 2 and 6, between 2 and 5, between 3 and 7) are 25 % or greater (e.g., 50 % or greater and 90 % or greater) by the weight of the fragrance. Those skilled in the art will appreciate that many fragrances can be created employing various solvents and fragrance ingredients. The use of relatively low to intermediate ClogP fragrance ingredients will result in fragrances that are suitable for encapsulation. These fragrances are generally water-insoluble, to be delivered through the capsule systems of this invention onto consumer products in different stages such as damp and dry fabric. Without encapsulation, the free fragrances would normally have evaporated or dissolved in water during use, e.g., wash. Whilst high logP materials have excellent encapsulation properties they are generally well delivered from a regular (non-

encapsulated) fragrance in a liquid fabric conditioning composition. Such fragrance chemicals would generally only need encapsulation for overall fragrance character purposes, very long-lasting fragrance delivery, or overcoming incompatibility with the liquid fabric conditioning composition, *e.g.*, fragrance materials that would otherwise be instable, cause thickening or discoloration of the product or otherwise negatively affect desired consumer product properties.

[0088] The active material to be encapsulated can be dispersed in aqueous solutions in the absence/presence of polymers, pre-condensates, surfactants, scavengers and the like prior to formation of capsules. In certain embodiments, the capsules include formaldehyde scavengers, which can be used from effective trace amounts up to 100 times the stoichiometric amount. The stoichiometric amount is the amount of scavenger required to theoretically bind or react all the formaldehyde added in the form of an aminoplast crosslinker (bound and free formaldehyde). This amount of scavenger can be added either to the slurry or afterward to the final product formulation. For instance, an unscavenged slurry can be added to the formulation, followed by a certain amount of scavenger. The particular quantity of a formaldehyde-based cross-linker that is used to create the capsule slurry contains a percentage of free formaldehyde and bound formaldehyde. The total combined moles of free and bound formaldehyde will determine the amount of moles of scavenger that is needed to react with all the formaldehyde. To drive this reaction to completion, a 10X molar excess of scavenger is used, preferably a 5X molar excess of scavenger. By moles here is meant moles of scavenging groups. Therefore, if the scavenger molecule is multifunctional (*i.e.*, polymeric) less moles of this molecule need to be added.

[0089] The minimum level of scavenger required is the amount that removes only the free formaldehyde in the slurry. This level is determined analytically. The minimum amount of moles of scavenger required is equal to the moles of analytically determined formaldehyde (1:1). Exemplary formaldehyde scavengers include β -dicarbonyl compounds; mono or di-amide scavengers; amines that form imines by reaction with formaldehyde; and formaldehyde reducers and sulfur containing compounds, such as those disclosed in US 2009/0258042.

[0090] Useful β -dicarbonyl compounds react with formaldehyde. Examples include, but are not limited to, acetoacetamide (BKB; Eastman), ethyl acetoacetate (EAA;

Eastman), N,N-dimethyleneacetamide (DMAA; Eastman), acetoacetone, dimethyl-1,3-acetonedicarboxylate, 1,3-acetonedicarboxylic acid, malonic acid, resorcinol, 1,3-cyclohexadione, barbituric acid, 5,5-dimethyl-1,3-cyclohexanedione (dimedone), 2,2-dimethyl-1,3-dioxane-4,6-dione (Meldrum's acid), salicylic acid, methyl acetoacetate (MAA; Eastman), ethyl-2-methyl acetoacetate, 3-methyl-acetoacetone, dimethyl malonate, diethyl malonate, 1,3-dimethyl barbituric acid, resorcinol, phloroglucinol, orcinol, 2,4-dihydroxy benzoic acid, 3,5-dihydroxy benzoic acid, malonamide and β -dicarbonyl scavengers listed in US 5,194,674 and US 5,446,195, as well as in Tomasino, et al. (1984) *Textile Chemist and Colorist* Vol. 16, No. 12.

[0091] Examples of useful mono- and di-amide scavengers are urea, ethylene urea, propylene urea, epsilon-caprolactam, glycouril, hydantoin, 2-oxazolidinone, 2-pyrrolidinone, uracil, barbituric acid, thymine, uric acid, allantoin, polyamides, 4,5-dihydroxyethylene urea, monomethylol-4-hydroxy-4-methoxy-5,5-dimethyl propylurea, nylon 2-hydroxyethyl ethylene urea (SR-511, SR-512; Sartomer), 2-hydroxyethyl urea (HYDROVANCE; National Starch), L-citrulline, biotin, N-methyl urea, N-ethyl urea, N-butyl urea, N-phenyl urea, 4,5-dimethoxy ethylene urea and succinimide.

[0092] Amines useful as scavengers include, but are not limited to, poly(vinyl amine) (LUPAMIN; BASF), arginine, lysine, asparagines, proline, tryptophan, 2-amino-2-methyl-1-propanol (AMP); proteins such as casein, gelatin, collagen, whey protein, soy protein, and albumin; melamine, benzoguanamine, 4-aminobenzoic acid (PABA), 3-aminobenzoic acid, 2-aminobenzoic acid (anthranilic acid), 2-aminophenol, 3-aminophenol, 4-aminophenol, creatine, 4-aminosalicylic acid, 5-aminosalicylic acid, methyl anthranilate, methoxylamine HCl, anthranilamide, 4-aminobenzamide, p-toluidine, p-anisidine, sulfanilic acid, sulfanilamide, methyl-4-aminobenzoate, ethyl-4-aminobenzoate (benzocain), beta-diethylaminoethyl-4-aminobenzoate (procain), 4-aminobenzamide, 3,5-diaminobenzoic acid and 2,4-diaminophenol. Other amines as disclosed in US 2006/0248665 and US 6,261,483, and those mentioned in Tomasino, et al. (1984) *Textile Chemist and Colorist* Vol. 16, No. 12, are also contemplated by the present invention. Other preferred amines can be selected from a non-limiting list of 1,2-phenylenediamine, 1,3-phenylenediamine, and 1,4-phenylenediamine. In addition, aromatic amines, triamines, and aliphatic polyamine may also be used. Examples of these amines may include, but are not limited to, aniline, hexamethylenediamine, bis-

hexamethylenetriamine, triethylaminetriamine, poly(propyleneoxide)triamine, and poly(propyleneglycol)diamines. Hydrazines such as 2,4-dinitrophenylhydrazine can also be used as scavengers. They react with formaldehyde to give hydrazones. The reaction is pH-dependent and reversible.

[0093] Other than scavengers, one or more adjunct material may be added to the delivery system in the amount of from 0.01 weight % to 25 weight % (e.g., from 0.5 weight % to 10 weight %).

[0094] The adjunct material can be a solubility modifier, an antibacterial, a sunscreen active, an antioxidant, a malodor counteracting agent, a density modifier, a stabilizer, a viscosity modifier, a pH modifier, or any combination thereof. These modifiers can be present in the wall or core of the capsules, or outside the capsules in the delivery system of this invention. Preferably, they are in the core as a core modifier.

[0095] Nonlimiting examples of a solubility modifier include surfactants (e.g., SLS and Tween 80), acidic compounds (e.g., mineral acids such as sulfuric acid, hydrochloric acid, nitric acid, and phosphoric acid, and carboxylic acids such as acetic acid, citric acid, gluconic acid, glucoheptonic acid, and lactic acid), basic compounds (e.g., ammonia, alkali metal and alkaline earth metal hydroxides, primary, secondary, or tertiary amines, and primary, secondary, or tertiary alkanolamines), ethyl alcohol, glycerol, glucose, galactose, inositol, mannitol, glactitol, adonitol, arabitol, and amino acids.

[0096] Exemplary antibacterials include bisguanidines (e.g., chlorhexidine digluconate), diphenyl compounds, benzyl alcohols, trihalocarbanilides, quaternary ammonium compounds, ethoxylated phenols, and phenolic compounds, such as halo-substituted phenolic compounds, like PCMX (i.e., p-chloro-m-xyleneol), triclosan (i.e., 2, 4, 4' -trichloro-2' hydroxy-diphenylether), thymol, and triclocarban.

[0097] Suitable sunscreen actives include oxybenzone, octylmethoxy cinnamate, butylmethoxy dibenzoyl ethane, p-aminobenzoic acid and octyl dimethyl-p-aminobenzoic acid.

[0098] Examples of antioxidants include beta-carotene, vitamin C (Ascorbic Acid) or an ester thereof, vitamin A or an ester thereof, vitamin E or an ester thereof, lutein or an ester thereof, lignan, lycopene, selenium, flavonoids, vitamin-like antioxidants such as coenzyme Q10 (CoQ10) and glutathione, and antioxidant enzymes such as superoxide dismutase (SOD), catalase, and glutathione peroxidase.

[0099] Malodor counteracting agents include, but not limited to, an α,β -unsaturated carbonyl compounds including but not limited to those disclosed in US 6,610,648 and EP 2,524,704, amyl cinnamaldehyde, benzophenone, benzyl benzoate, benzyl isoeugenol, benzyl phenyl acetate, benzyl salicylate, butyl cinnamate, cinnamyl butyrate, cinnamyl isovalerate, cinnamyl propionate, decyl acetate, ethyl myristate, isobutyl cinnamate, isoamyl salicylate, phenethyl benzoate, phenethyl phenyl acetate, triethyl citrate, tripropylene glycol n-butyl ether, isomers of bicyclo[2.2.1]hept-5-ene-2-carboxylic acid, ethyl ester, nano silver, and zinc undecenylate. More suitable malodor counteracting agents are polymers containing an α -keto, benzaldehyde, or α,β -unsaturated carbonyl moiety, such as those described in US Application Publications 2012/0294821, 2013/0101544 and 2013/0101545.

[00100] The density of the capsule slurry and/or the oil core can be adjusted so that the capsule compositions have a substantially uniform distribution of the capsules using known density modifiers or technologies such as those described in Patent Application Publications WO 2000/059616, EP 1 502 646, and EP 2 204 155. Suitable density modifiers include hydrophobic materials and materials having a desired molecular weight (e.g., higher than 12,000), such as silicone oils, petrolatums, vegetable oils, especially sunflower oil and rapeseed oil, and hydrophobic solvents having a desired density (e.g., less than 1,000 Kg/m³ at 25°C, such as limonene and octane).

[00101] In some embodiments, a stabilizer (e.g., a colloidal stabilizer) is added to capsule compositions to stabilize the emulsion and/or capsule slurry. Examples of colloidal stabilizers are polyvinyl alcohol, cellulose derivatives such hydroxyethyl cellulose, polyethylene oxide, copolymers of polyethylene oxide and polyethylene or polypropylene oxide, or copolymers of acrylamide and acrylic acid.

[00102] Viscosity control agents (e.g., suspending agents), which may be polymeric or colloidal (e.g., modified cellulose polymers such as methylcellulose, hydroxyethylcellulose, hydrophobically modified hydroxyethylcellulose, and cross-linked acrylate polymers such as Carbomer, hydrophobically modified polyethers) can be included in capsule compositions described above, either in the oil core or capsule wall, or in the capsule slurry outside the capsules. Optionally, silicas, either hydrophobic or hydrophilic, can be included at a concentration from 0.01 % to 20 %, more preferable from 0.5 % to 5 %, by the weight of the capsule compositions. Examples of hydrophobic

silicas include silanols, surfaces of which are treated with halogen silanes, alkoxysilanes, silazanes, and siloxanes, such as SIPERNAT D17, AEROSIL R972 and R974 available from Degussa. Exemplary hydrophilic silicas are AEROSIL 200, SIPERNAT 22S, SIPERNAT 50S (available from Degussa), and SYLOID 244 (available from Grace Davison).

[00103] One or more humectants are optionally included to hold water in the capsule compositions for a long period of time. They constitutes from 0.01% to 25% (e.g., 1% to 5%) by weight of the capsule composition. Examples include glycerin, propylene glycol, alkyl phosphate esters, quaternary amines, inorganic salts (e.g., potassium polymetaphosphate, sodium chloride, etc.), polyethylene glycols, and the like.

[00104] Further suitable humectants, as well as viscosity control/suspending agents, are disclosed in US 4,428,869, 4,464,271, 4,446,032, and 6,930,078. Details of hydrophobic silicas as a functional delivery vehicle of active materials other than a free flow/anticaking agent are disclosed in US 5,500,223 and 6,608,017.

[00105] In some embodiments, one or more pH modifiers are included in a capsule composition to adjust the pH value of the capsule slurry and/or the oil core. The pH modifiers can also assist in the formation of capsule walls by changing the reaction rate of the crosslinking reactions that form the capsule walls. Exemplary pH modifiers include metal hydroxides (e.g., LiOH, NaOH, KOH, and Mg(OH)₂), metal carbonates and bicarbonates (CsCO₃, Li₂CO₃, K₂CO₃, NaHCO₃, and CaCO₃), metal phosphates/hydrogen phosphates/dihydrogen phosphates, metal sulfates, ammonia, mineral acids (HCl, H₂SO₄, H₃PO₄, and HNO₃), carboxylic acids (e.g., acetic acid, citric acid, lactic acid, benzoic acid, and sulfonic acids), and amino acids.

[00106] The capsule compositions of this invention can also include one or more non-confined unencapsulated active materials from 0.01 weight % to 50 weight %, more preferably from 5 weight % to 40 weight %.

[00107] The level of solvent materials, particles, adjuncts, or core modifiers can be greater than 10% (e.g., greater than 30% and greater than 70%). In addition to the solvent, it is preferred that a fragrance having a weight-averaged ClogP of 2.5 and greater (e.g., 3 or greater, 2.5 to 7, and 2.5 to 5) is employed. The weight-averaged ClogP is calculated as follows:

$$\text{ClogP} = \{ \text{Sum} [(W_i)(\text{ClogP})_i] \} / \{ \text{Sum } W_i \},$$

in which W_i is the weight fraction of each fragrance ingredient and $(\text{ClogP})_i$ is the ClogP of that fragrance ingredient.

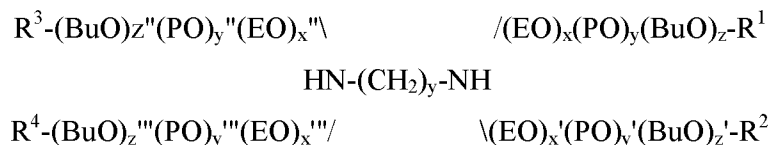
[00108] As an illustration, it is preferred that greater than 60 weight percent, preferably greater than 80 and more preferably greater than 90 weight percent of the fragrance chemicals have ClogP values of greater than 3.3, preferably greater than 4 and even more preferably greater than 4.5. Those with skill in the art will appreciate that many formulations can be created employing various solvents and fragrance chemicals. The use of a high level of high ClogP fragrance chemicals will likely require a lower level of hydrophobic solvent than fragrance chemicals with lower ClogP to achieve similar performance stability. As those with skill in the art will appreciate, in a highly preferred embodiment, high ClogP fragrance chemicals and hydrophobic solvents comprise greater than 80, preferably more than 90 and even more preferably greater than 95 weight percent of the fragrance composition. As discussed above, specific ClogP values may be measured between candidate solvents and water for the fragrance materials to be included in the core. In this way, an optimum solvent choice may be made. In fact, since most fragrances will have many ingredients, it may be preferable to measure the partitioning of a specific fragrance blend in solvent and water in order to determine the effect of any material interactions.

[00109] Optionally, an emulsifier (*i.e.*, nonionic such as polyoxyethylene sorbitan monostearate (e.g., TWEEN 60), anionic such as sodium oleate, zwitterionic such as lecithins) from 0.01 weight % to 25 weight %, more preferably from 5 weight % to 10 weight % can be included.

[00110] A capsule deposition aid (*i.e.*, cationic starches such as Hi-CAT CWS42, cationic guar gums such as Jaguar C-162, cationic amino resins, cationic urea resins, hydrophobic quaternary amines, etc.) from 0.01 weight % to 25 weight %, more preferably from 5 weight % to 20 weight % can be included. The capsule deposition aid can be added during the preparation of the capsules or it can be added after the capsules have been made. More exemplary deposition aids are described in US Patent Application Publications 2013/0330292, 2013/0337023, and 2014/0017278.

[00111] Additional polymeric core modifiers are also contemplated. It has been found that the addition of hydrophobic polymers to the core can also improve stability by slowing diffusion of the active material from the core. The level of polymer is normally

less than 80% of the core by weight, preferably less than 50%, and more preferably less than 20%. The basic requirement for the polymer is that it be miscible or compatible with the other components of the core, namely the active material and other solvent. Preferably, the polymer also thickens or gels the core, thus further reducing diffusion. These additional polymeric core modifiers include copolymers of ethylene; copolymers of ethylene and vinyl acetate (ELVAX polymers by DOW Corporation); copolymers of ethylene and vinyl alcohol (EVAL polymers by Kuraray); ethylene/acrylic elastomers such as VALNAC polymers by Dupont; polyvinyl polymers, such as polyvinyl acetate; alkyl-substituted cellulose, such as ethyl cellulose (ETHOCEL made by DOW Corporation) and hydroxypropyl celluloses (KLUCCEL polymers by Hercules); cellulose acetate butyrate available from Eastman Chemical; polyacrylates (*e.g.*, AMPHOMER, DEMACRYL LT and DERMACRYL 79, made by National Starch and Chemical Company, the AMERHOLD polymers by Amerchol Corporation, and ACUDYNE 258 by ISP Corporation); copolymers of acrylic or methacrylic acid and fatty esters of acrylic or methacrylic acid such as INTELIMER POLYMERS made by Landec Corporation (see also U.S. Pat. Nos. 4,830,855, 5,665,822, 5,783,302, 6,255,367 and 6,492,462); polypropylene oxide; polybutylene oxide of poly(tetrahydrofuran); polyethylene terephthalate; polyurethanes (DYNAM X by National Starch); alkyl esters of poly(methyl vinyl ether); maleic anhydride copolymers, such as the GANTREZ copolymers and OMNIREZ 2000 by ISP Corporation; carboxylic acid esters of polyamines, *e.g.*, ester-terminated polyamides (ETPA) made by Arizona Chemical Company; polyvinyl pyrrolidone (LUVISKOL series of BASF); block copolymers of ethylene oxide, propylene oxide and/or butylenes oxide including, *e.g.*, PLURONIC and SYNPERONIC polymers/dispersants by BASF. Another class of polymers include polyethylene oxide-co-propyleneoxide-co-butylene oxide polymers of any ethylene oxide/propylene oxide/butylene oxide ratio with cationic groups resulting in a net theoretical positive charge or equal to zero (amphoteric). The general structure is:



where R^1 , R^2 , R^3 , and R^4 are independently H or any alkyl or fatty alkyl chain group. Examples of such polymers are the commercially known as TETRONICS by BASF Corporation.

[00112] Sacrificial core ingredients can also be included. These ingredients are designed to be lost during or after manufacture and include, but are not limited to, highly water soluble or volatile materials.

[00113] In addition to the capsules and adjunct materials described above, the capsule compositions of this invention can contain one or more other delivery compositions such as polymer-assisted delivery compositions (see US 8,187,580), fiber-assisted delivery compositions (US 2010/0305021), cyclodextrin host guest complexes (US 6,287,603 and US 2002/0019369), pro-fragrances (WO 2000/072816 and EP 0 922 084), membrane delivery systems (US 4,948,047), and any combination thereof.

[00114] As used herein olfactory effective amount is understood to mean the amount of compound in the delivery system the individual components will contribute to its particular olfactory characteristics, but the olfactory effect of the delivery system will be the sum of the effects of each of the individual components. Thus, the capsule delivery systems of this invention can be used to alter the aroma characteristics of a consumer product, e.g., a fine perfume, by modifying the olfactory reaction contributed by another ingredient in the consumer product. The amount will vary depending on many factors including other ingredients, their relative amounts and the effect that is desired.

[00115] In accordance with some embodiments of this invention, the capsules can be cured at a temperature in the range of, e.g., 55°C to 130°C (e.g., 55°C to 90°C, 55°C to 75°C, and 90°C to 130°C) for 1 minute to 10 hours (e.g., 0.1 hours to 5 hours, 0.2 hours to 4 hours and 0.5 hours to 3 hours). A skilled person in the art can determine, without undue experiments, the curing temperature, duration, and the heating rate.

[00116] Not to be bound by any theory, it is believed that there is a direct relationship between higher cure temperature and less leaching of active material from the capsule. In accordance with one embodiment, the capsules are cured at a temperature at or above 100°C. The retention capabilities of the capsules are thus improved. In another embodiment, the capsules are cured at or above 110°C. In still another embodiment, the capsules are cured at or above 120°C.

[00117] To obtain capsules with more leaching of the active material, certain embodiments of this invention provide for a cure temperature of less than 100°C. In some embodiments, the cure temperature is at or less than 90°C. In other embodiments, the cure temperature is at or less than 80°C.

[00118] In one embodiment, the capsules are heated to a target cure temperature at a linear rate of 0.5 to 2 °C per minute (e.g., 1 to 5 °C per minute, 2 to 8 °C per minute, and 2 to 10°C per minute) over a period of 1 to 60 minutes (e.g., 1 to 30 minutes). The following heating methods may be used: conduction for example via oil, steam radiation via infrared, and microwave, convection via heated air, steam injection and other methods known by those skilled in the art. The target cure temperature used herein refers to the minimum temperature in degrees Celsius at which the capsules may be cured to retard leaching.

[00119] Useful capsules in this invention preferably have a size in the range of from 0.01 to 1000 microns (e.g., 0.5 to 1000 microns, 1 to 200 microns, 0.5 to 150 microns, 2 to 50 microns, 5 to 25 microns, and 2 to 15 microns). The capsule distribution can be narrow, broad, or multi-modal.

[00120] In some embodiments, the capsule is purified before formulated into the liquid fabric conditioning composition. Purification can be achieved by washing with water or a salt solution, diafiltration, and centrifugation. Suitable diafiltration processes are those described in US Patent Application Publication 2014/0134242 and Sheth et al., J. of Membr. Sci. 2003, 211.2, 251-61. The capsule slurry can also be washed with water until a neutral pH in the capsule slurry is achieved. For the purposes of the present invention, the capsule compositions can be washed using any conventional method including the use of a separatory funnel, filter paper, centrifugation and the like. The capsule suspension can be washed one, two, three, four, five, six, seven, eight, nine, ten or more times until a neutral pH, i.e., $\text{pH } 7 \pm 0.5$, is achieved. The pH of the purified capsules can be determined using any conventional method including, but not limited to pH paper, pH indicators, or a pH meter.

[00121] A capsule suspension of this invention is “purified” in that it is 80%, 90%, 95%, 97%, 98% or 99% homogeneous to capsules. In accordance with the present invention, purity is achieved by washing the capsules until a neutral pH is achieved,

which is indicative of removal of unwanted impurities and/or starting materials, e.g., polyisocyanate, cross-linking agent and the like.

[00122] In certain embodiments of this invention, the purification of the capsules includes the additional step of adding a salt to the capsule suspension prior to the step of washing the capsule suspension with water. Exemplary salts of use in this step of the invention include, but are not limited to, sodium carbonate, sodium bicarbonate, sodium chloride, potassium carbonate, potassium bicarbonate, potassium chloride, and bi-sulphite salts.

[00123] All parts, percentages and proportions refer to herein and in the claims are by weight unless otherwise indicated.

[00124] The values and dimensions disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such value is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a value disclosed as "50%" is intended to mean "about 50%."

[00125] The invention is described in greater detail by the following non-limiting examples.

[00126] All publications recited herein are incorporated by reference in their entirety.

EXAMPLE 1

[00127] A fabric conditioning composition of this invention, *i.e.*, Sample 1, was prepared using Capsule Composition Eden PU described below.

[00128] Eden PU was prepared following the procedure described below. Also see US Patent 8,299,011.

[00129] A fragrance emulsion was first prepared by mixing 96 g of a fragrance, Eden (International Flavors and Fragrance, Union Beach, NJ), 24 g of NEOBEE oil (Stepan, Chicago, IL) and 9.6 g of isocyanate monomer, Lupranate®M20 (BASF corporation, Wyandotte, Mich., USA) to form an oil phase. In a separate beaker, a 3% surfactant solution (160 g) was prepared by dissolving 4.8 g of Morwet D-425 (Akzo Nobel, Fort Worth, Tex., USA) in water. The oil phase was then emulsified into the surfactant solution to form a fragrance emulsion under shearing (Ultra Turrax®, T25 Basic, IKA® WERKE) at 6500 rpm for two minutes.

[00130] To the fragrance emulsion thus prepared was added 10.8 g of 40% hexamethylenediamine (HMDA) (INVISTA, Wichita, Kans., USA) under constant mixing with an overhead mixer. Formation of polyurea ("PU") capsule was immediately visible by optical microscopy. The mixer speed was reduced after the addition of HMDA was complete. The capsule slurry was cured at 55 °C for 2 hours to obtain Capsule Composition Eden PU.

[00131] Eden PU thus prepared typically contains 0.4 to 1% free HMDA, which can be measured by Liquid chromatography–mass spectrometry (LC-MS) or Gas chromatography (GC).

[00132] Sample 1 contains an unfragranced fabric softener base, 0.9% of a fragrance oil, 0.2% neat oil equivalent ("NOE") of Eden PU, and 0.2% NOE of a poly(melamine-formaldehyde) ("MF") capsule. The fabric softener base, commercially available from Unilever under the brand name Comfort, has 10.5% of a fabric conditioning agent. The fragrance oil is commercially available from International Flavors and Fragrances under the name Rising Legend. And the MF capsule is Thunder, commercially available also from International Flavors and Fragrances.

[00133] To prepare Sample 1, the following ingredients were mixed until homogeneous: 977.6 g of unfragranced softener base, 9 g of Rising Legend, 7.14 g of Thunder, and 6.25 g of Eden PU.

Comparative sample

[00134] For comparison, a comparative fabric conditioning composition, *i.e.*, Comparative 1', was also prepared using the same ingredients as Sample 1 except that 7.14 g of Eden (a MF capsule commercially available from International Flavors and Fragrances) was used instead of Eden PU.

Viscosity stability evaluation

[00135] Sample 1 and Comparative 1' were evaluated for their viscosity stabilities stored at 37 °C and 40 °C for 4, 8, and 12 weeks.

[00136] Each viscosity was measured on a Physica ASC32-MCR300 with a concentric cylinder at a constant shear rate of 20 second⁻¹ for a period of 3 minutes under 25 °C.

[00137] The viscosity results are shown in Table 2 below.

Table 2

Fabric Softener	Viscosity (mPa.s) at 20 second ⁻¹						
	initial	4 weeks		8 weeks		12 weeks	
		37 °C	40 °C	37 °C	40 °C	37 °C	40 °C
Sample 1	42	39	54	38	55	40	69
Comparative 1'	45	73	128	77	162	89	220

[00138] Unexpectedly, Sample 1 containing stabilizing agent HMDA has a much greater viscosity stability than Comparative 1' without a stabilizing agent during the extended period of storage at the two temperatures in this evaluation.

EXAMPLE 2

[00139] A second fabric conditioning composition of this invention, *i.e.*, Sample 2, was prepared using Capsule Composition LL Cloud PU described below.

[00140] Capsule Composition LL Cloud PU was prepared following the same procedure for Eden PU described in Example 1 above except that fragrance LL Cloud (commercially available from International Flavors and Fragrance, Union Beach, NJ), instead of Eden, was used.

[00141] Capsule Composition LL Cloud PU thus prepared typically contains 0.4 to 1% free HMDA, which can be measured by an instrument such as LC/MS and GC.

[00142] Sample 2 of this invention contains an unfragranced model fabric softener base, 0.76% of a fragrance oil, 0.3% NOE of LL Cloud PU.

[00143] The unfragranced model fabric softener has 12% esterquat (Rewoquat WE18, a fabric conditioning agent commercially available from Evonik). The fragrance oil is Spring Equinox commercially available from International Flavors and Fragrances.

Comparative 2'

[00144] For comparison, a comparative fabric conditioning composition, *i.e.*, Comparative 2', was also prepared using the same ingredients as Sample 2 except that 0.3% NOE Jillz (a MF capsule commercially available from International Flavors and Fragrances) was used instead of LL Cloud PU.

Viscosity stability evaluation

[00145] Sample 2 and Comparative 2', along with the unfragranced model fabric softener, were evaluated for their viscosity stabilities stored at 37 °C and 40 °C for 4, 8,

and 12 weeks following the same procedure described in Example 1 above. The viscosity results are shown in Table 3 below.

Table 3

Fabric Softener	Viscosity (mPa.s) at 20 second ⁻¹						
	initial	4 Weeks		8 Weeks		12 Weeks	
		37 °C	40 °C	37 °C	40 °C	37 °C	40 °C
Sample 2	57	49	50	44	49	46	61
Model Softener	56	58	70	66	68	73	138
Comparative 2'	87	79	92	75	95	70	122

[00146] Unexpectedly, Sample 2 containing stabilizing agent HMDA has a much greater viscosity stability than the model softener and Comparative 2', both of which do not contain a stabilizing agent.

EXAMPLE 3

[00147] A third fabric conditioning composition of this invention, i.e., Sample 3, was prepared using Capsule Composition Greenfields PU described below.

[00148] Greenfields PU was prepared following the same procedure for Eden PU described in Example 1 except that fragrance Greenfields (commercially available from International Flavors and Fragrance, Union Beach, NJ), instead of Eden, was used.

[00149] Capsule Composition Greenfields PU thus prepared typically contains 0.4 to 1% free HMDA, which can be measured by an instrument such as LC/MS and GC.

[00150] Sample 3 of this invention contains 0.6% NOE of Greenfields PU and an unfragranced model fabric softener base having 20% of a fabric conditioning agent and.

Comparative 3'

[00151] For comparison, a comparative fabric softener, i.e., Comparative 3', was also prepared using the same ingredients as Sample 3 except that 0.6% NOE Greenfields MF (a MF capsule commercially available from International Flavors and Fragrances) was used instead of Greenfields PU.

Viscosity stability evaluation

[00152] Sample 3 and Comparative 3', along with the unfragranced model fabric softener, were evaluated for their viscosity stabilities stored at 37 °C for 4 and 8 weeks following the same procedure described in Example 1 above.

[00153] The viscosity results are shown in Table 4 below.

Table 4.

Compositions	Viscosity (mPa.s) at 20 second ⁻¹		
Sample 3	90	99	163
Unfragranced	80	102	272
Comparative 3'	98	129	219

[00154] Unexpectedly, Sample 3 containing stabilizing agent HMDA has a much greater viscosity stability than the unfragranced model conditioning composition and Comparative 3', both of which do not contain a stabilizing agent.

EXAMPLES 4-11

[00155] Five more fabric conditioning compositions of this invention, i.e., Samples 4-11, were prepared by adding 40% HMDA solution to a fabric conditioning base containing 12% esterquat as the conditioning agent. A comparative sample, i.e., Comparative 4', were used for comparison.

[00156] More specifically, the ingredient(s) of each sample/comparative are shown below (in addition to the conditioning agent):

Sample 4: 80 ppm HMDA by weight of the liquid fabric composition

Sample 5: 160 ppm HMDA

Sample 6: 320 ppm HMDA

Sample 7: 640 ppm HMDA

Comparative 4': 0.3% NOE Blueday MF (commercially available from International Flavors and Fragrances, Union Beach, NJ)

Sample 8: 80 ppm HMDA and 0.3% NOE Blueday MF

Sample 9: 160 ppm HMDA and 0.3 NOE Blueday MF

Sample 10: 320 ppm HMDA and 0.3 NOE Blueday MF

Sample 11: 160 ppm HMDA and 0.3 NOE Blueday MF

Viscosity stability evaluation

[00157] Samples 4-11, unfragranced conditioning base, and Comparative 4' were evaluated for their viscosity stabilities stored at 20 °C for 2 and 4 weeks following the same procedure described in Example 1 above.

[00158] The viscosity results are shown in Table 5 below.

Table 5

Composition	Viscosity (mPa.s) at 20 second ⁻¹		
	Initial	2 weeks	4 weeks
Sample 4	171	155	149
Sample 5	157	138	126
Sample 6	134	109	94
Sample 7	114	87	73
Comparative 4'	168	172	176
Sample 8	150	142	135
Sample 9	137	125	115
Sample 10	122	105	92
Sample 11	107	88	77

[00159] Unexpectedly, Samples 4-7 containing stabilizing agent HMDA from 80 to 640 ppm showed viscosity stability and, compared to Comparative 4' containing no HMDA, Samples 8-11 showed greater viscosity stability, each of which contains HMDA at a various concentration from 80 to 640 ppm.

OTHER EMBODIMENTS

[00160] All of the features disclosed in this specification may be combined in any combination. Each feature disclosed in this specification may be replaced by an alternative feature serving the same, equivalent, or similar purpose. Thus, unless expressly stated otherwise, each feature disclosed is only an example of a generic series of equivalent or similar features.

[00161] Indeed, to achieve the purpose of preparing a stable liquid fabric conditioning composition, one skilled in the art can choose a suitable fabric conditioning agent and stabilizing agent and determine their concentrations.

[00162] From the above description, a skilled artisan can easily ascertain the essential characteristics of the present invention, and without departing from the spirit and scope thereof, can make various changes and modifications of the invention to adapt it to various usages and conditions. Thus, other embodiments are also within the claims.

WHAT IS CLAIMED IS:

1. A liquid fabric conditioning composition comprising:
1 to 30% of a fabric conditioning agent,
0.1 to 30% of a capsule having an oil core and a capsule wall, the oil core containing an active material and the capsule wall encapsulating the oil core, and
20 ppm or greater of a stabilizing agent,
wherein the active material is a fragrance, a malodor counteractant, or a combination thereof; the capsule wall is formed of polyurea, polyurethane, polyacrylate, polyacrylamide, poly(acrylate-co-acrylamide), starch, silica, gelatin and gum Arabic, poly(melamine-formaldehyde), poly(urea-formaldehyde), or a combination thereof; and the stabilizing agent is a multi-functional amine, a mono-functional amine, an amino acid, a polymer mixture, or a combination thereof, the polymer mixture containing a first polymer selected from the group consisting of polyvinylpyrrolidone, polyvinylpyridine-N-oxide, polylysine, and polyvinyl imidazolinium, and a second polymer.
2. The liquid fabric conditioning composition of claim 1, wherein the stabilizing agent, being at a concentration of 50 ppm to 2%, is hexamethylene diamine, hexaethylenediamine, ethylenediamine, 1,3-diaminopropane, 1,4-diamino-butane, diethylenetriamine, pentaethylenhexamine, bis(3-aminopropyl)amine, bis(hexanethylene)triamine, tris(2-aminoethyl)amine, triethylene-tetramine, N,N'-bis(3-aminopropyl)-1,3-propanediamine, tetraethylenepentamine, branched polyethylenimine, chitosan, nisin, gelatin, 1,3-diamino-guanidine, 1,1-dimethylbiguanide, guanidine, arginine, lysine, ornithine, histidine, amino-2-methyl-1-propanol, or a combination thereof.
3. The liquid fabric conditioning composition of claim 2, wherein the stabilizing agent is hexamethylenediamine, amino-2-methyl-1-propanol, lysine, arginine, histidine, or a combination thereof at a concentration of 50 ppm to 1%.
4. The liquid fabric conditioning composition of claim 1, wherein the fabric conditioning agent is present at a concentration of 2 to 25%.

5. The liquid fabric conditioning composition of claim 4, wherein the fabric conditioning agent is present at a concentration of 4 to 15%.

6. The liquid fabric conditioning composition of claim 1, wherein the composition contains 0.15 to 15% of the capsule, the capsule contains 35 to 99.5% of the core oil and 0.5 to 65% of the capsule wall, and the capsule wall is formed of polyurea or polyurethane.

7. The liquid fabric conditioning composition of claim 6, wherein the composition contains 0.5 to 10 wt% of the capsule, the capsule contains 50 to 98% of core oil and 2 to 50% of capsule wall.

8. The liquid fabric conditioning composition of claim 7, wherein the fabric conditioning agent is present at a concentration of 2 to 25%, and the stabilizing agent is at a concentration of 50 ppm to 2% and selected from the group consisting of hexamethylenediamine, amino-2-methyl-1-propanol, lysine, arginine, histidine, or a combination thereof.

9. The liquid fabric conditioning composition of claim 7, wherein the fabric conditioning agent is present at a concentration of 4 to 15%, and the stabilizing agent is hexamethylenediamine at a concentration of 50 to 2000 ppm.

10. The liquid fabric conditioning composition of claim 1, wherein the composition contains 0.15 to 15% of the capsule, the capsule contains 35 to 99.5% of the core oil and 0.5 to 65% of the capsule wall, and the capsule wall is formed of poly(melamine-formaldehyde).

11. The liquid fabric conditioning composition of claim 10, wherein the composition contains 0.5 to 10% of the capsule, the capsule contains 50 to 98% of core oil and 2 to 50% of capsule wall.

12. The liquid fabric conditioning composition of claim 10, wherein the fabric conditioning agent is present at a concentration of 2 to 25%, and the stabilizing agent is present at a concentration of 50 ppm to 2% and selected from the group consisting of hexamethylenediamine, amino-2-methyl-1-propanol, lysine, arginine, histidine, or a combination thereof.

13. The liquid fabric conditioning composition of claim 12, wherein the fabric conditioning agent is present at a concentration of 4 to 15%, and the stabilizing agent is hexamethylenediamine at a concentration of 50 to 2000 ppm.

14. The liquid fabric conditioning composition of claim 1, wherein the composition contains a second, third, or fourth capsule, each of which is different from each other in wall material, amount of wall material, ratio of wall material to oil core, core modifier, scavenger, active material, curing temperature, heating rate, curing time or a combination thereof.

15. The liquid fabric conditioning composition of claim 1, further comprising a malodor counteractant.

16. The liquid fabric conditioning composition of claim 15, wherein the malodor counteractant is 1-cyclohexylethan-1-yl butyrate, 1-cyclohexylethan-1-yl acetate, 1-cyclohexylethan-1-ol, 1-(4'-methylethyl)cyclohexylethan-1-yl propionate, 2'-hydroxy-1'-ethyl(2-phenoxy)acetate, or a combination thereof.

17. The liquid fabric conditioning composition of claim 1, further comprising a deposition aid that is an anionic, cationic, nonionic or zwitterionic polymer.

18. The liquid fabric conditioning composition of claim 17, wherein the deposition aid is polyquaternium-6, polyquaternium-47, polyquaternium-53, polyvinylamine, poly(vinsylamine-co-vinylformamide), or a combination thereof.

19. A liquid fabric conditioning composition comprising 1 to 30% of a fabric conditioning agent and 20 ppm or greater of a stabilizing agent, the stabilizing agent being a multi-functional amine, a mono-functional amine, or an amino acid.

20. The liquid fabric conditioning composition of claim 19, wherein the stabilizing agent, being at a concentration of 50 ppm to 2%, is hexamethylenediamine, amino-2-methyl-1-propanol, lysine, arginine, histidine, or a combination thereof.

21. The liquid fabric conditioning composition of claim 20, wherein the stabilizing agent is hexamethylenediamine at a concentration of 50 to 2000 ppm.

22. The liquid fabric conditioning composition of claim 20, wherein the fabric conditioning agent is present at a concentration of 2 to 25%.

23. The liquid fabric conditioning composition of claim 22, wherein the fabric conditioning agent is present at a concentration of 4 to 15%.

24. The liquid fabric conditioning composition of claim 23, wherein the stabilizing agent is present at a concentration of 50 ppm to 2% and selected from the group consisting of hexamethylenediamine, amino-2-methyl-1-propanol, lysine, arginine, histidine, or a combination thereof.

25. The liquid fabric conditioning composition of claim 24, wherein the stabilizing agent is hexamethylenediamine at a concentration of 50 to 2000 ppm.

26. A method of stabilizing the viscosity of a liquid fabric conditioning composition, the method comprising the steps of:

providing a liquid fabric conditioning base containing 1-30% of a fabric conditioning agent,

mixing a stabilizing agent with the liquid fabric conditioning base, wherein the stabilizing agent, being at a concentration of 20 ppm or greater, is a multi-functional amine, mono-functional amine, or an amino acid.

27. The method of claim 26, wherein , wherein the stabilizing agent, being at a concentration of 50 ppm to 1%, is hexamethylenediamine, amino-2-methyl-1-propanol, lysine, arginine, histidine, or a combination thereof.

28. The method of claim 27, wherein the stabilizing agent is hexamethylenediamine at a concentration of 50 to 2000 ppm.

INTERNATIONAL SEARCH REPORT

International application No.

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A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - C11D 3/50, 1/835 (2016.01)

CPC - C11D 3/50, 3/502, 1/835

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: C11D 3/50, 1/835 (2016.01)

CPC: C11D 3/50, 3/502, 1/835

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

PatSeer (US, EP, WO, JP, DE, GB, CN, FR, KR, ES, AU, IN, CA, Other Countries (INPADOC), RU, AT, CH, TH, BR, PH), EBSCO, Google/Google Scholar, IP.com Keywords: fabric softener, oil core, capsule, fragrance, viscosity

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 8,026,206 B2 (SAJIC, B et al.) September 27, 2011; column 3, lines 40-50; column 4, lines 1-30	26
Y		27-28
Y	US 7,632,789 B2 (BRAIN, J et al.) December 15, 2009; column 2, lines 5-10, 40-51; column 4, lines 15-25; column 8, lines 30-40; column 12, lines 50-65; column 14, 35-60; column 17, lines 30-50	1-25
Y	US 2013/0337023 A1 (INTERNATIONAL FLAVORS & FRAGRANCES INC.) December 19, 2013; paragraphs [0004], [0012], [0019]-[0020], [0022]-[0023], [0026], [0067], [0088]-[0089], [0104]-[0105]	1-25, 27-28
Y	US 2004/0156742 A1 (MILAN, JB et al.) August 12, 2004; abstract	16

☐ Further documents are listed in the continuation of Box C.☐ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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"E" earlier application or patent but published on or after the international filing date	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"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)	"&" document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means	
"P" document published prior to the international filing date but later than the priority date claimed	

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