Canadian Intellectual Property Office

CA 3236455 A1 2023/06/08

(21) 3 236 455

(12) DEMANDE DE BREVET CANADIEN CANADIAN PATENT APPLICATION

(13) **A1**

(86) Date de dépôt PCT/PCT Filing Date: 2022/11/23

(87) Date publication PCT/PCT Publication Date: 2023/06/08

(85) Entrée phase nationale/National Entry: 2024/04/26

(86) N° demande PCT/PCT Application No.: US 2022/080398

(87) N° publication PCT/PCT Publication No.: 2023/102341

(30) Priorité/Priority: 2021/12/03 (US17/541,316)

(51) **CI.Int./Int.CI.** *C11D 1/14* (2006.01), *C11D 1/22* (2006.01), *C11D 1/37* (2006.01)

(71) **Demandeur/Applicant:**

THE PROCTER & GAMBLE COMPANY, US

(72) Inventeurs/Inventors:

STENGER, PATRICK CHRISTOPHER, US; AULTMAN, ERIN JULIETTE, US; LOUGHNANE, BRIAN JOSEPH, US; BECKS, VINCENT JOHN, US

(74) Agent: MBM INTELLECTUAL PROPERTY AGENCY

(54) Titre: COMPOSITIONS DETERGENTES LIQUIDES (54) Title: LIQUID DETERGENT COMPOSITIONS

(57) Abrégé/Abstract:

Liquid detergent compositions can include a first surfactant which is a mixture of surfactant isomers of Formula 1 and surfactants of Formula 2: CH_2 -X I Formula 1: CH_3 - $(CH_2)_m$ -CH- $(CH_2)_n$ - CH_3 6 \leq m \leq 11; 0 \leq n \leq 5; Formula 2: CH_3 - $(CH_2)_{m+n+3}$ -X wherein from about 50% to about 100% by weight of the first surfactant are isomers having m+n = 11; wherein between about 25% to about 50% of the mixture of surfactant isomers of Formula 1 have n = 0; wherein from about 0.001% to about 25% by weight of the first surfactant are surfactants of Formula 2; and wherein X is a hydrophilic moiety; and an encapsulate including a shell and a core, wherein the shell includes a polyacrylate and the core includes a perfume.





Date Submitted: 2024/04/26

CA App. No.: 3236455

Abstract:

Liquid detergent compositions can include a first surfactant which is a mixture of surfactant isomers of Formula 1 and surfactants of Formula 2: CH2—X I Formula 1:

CH3—(CH2)m—CH—(CH2)n—CH3 6 m 11; 0 n 5; Formula 2: CH3—(CH2)m+n+3—X wherein from about 50% to about 100% by weight of the first surfactant are isomers having m+n = 11; wherein between about 25% to about 50% of the mixture of surfactant isomers of Formula 1 have n = 0; wherein from about 0.001% to about 25% by weight of the first surfactant are surfactants of Formula 2; and wherein X is a hydrophilic moiety; and an encapsulate including a shell and a core, wherein the shell includes a polyacrylate and the core includes a perfume.

LIQUID DETERGENT COMPOSITIONS

FIELD OF THE INVENTION

Liquid detergent compositions which include a first surfactant and an encapsulate comprising a shell and a core, wherein the shell comprises a polyacrylate and the core comprises a perfume.

BACKGROUND OF THE INVENTION

Liquid detergent compositions are routinely used to wash substrates, like fabric. The formulation of a liquid detergent composition is a balance, among other things, of the ability to sufficiently clean the target substrate without damaging the substrate being cleaned. In addition, many consumers like laundered substrates to have a pleasant scent after washing. While some scent can be applied to the substrate through the washing process, it is sometimes not at a consumer preferred level. This can lead to the utilization of perfume technologies, like a perfume encapsulate to help further boost scent. Encapsulates can be difficult to efficiently deliver to a target substrate leading many to be washed away down the drain where their benefit cannot be utilized. Thus, it is beneficial to find and utilize materials in a formulation which can help deposit encapsulates onto a target substrate. As such, there is a need for materials which can be utilized in a liquid detergent composition which can help deposit encapsulates onto a target substrate.

20

25

5

10

15

SUMMARY OF THE INVENTION

Included herein, for example, is a liquid detergent composition comprising a) from about 1% to about 30%, by weight of the composition of a first surfactant consisting essentially of a mixture of surfactant isomers of Formula 1 and surfactants of Formula 2:

 $6 \le m \le 11$; $0 \le n \le 5$;

Formula 2: CH_3 — $(CH_2)_{m+n+3}$ —X

wherein from about 50% to about 100% by weight of the first surfactant are isomers having m+n=11; wherein between about 25% to about 50% of the mixture of surfactant isomers of Formula 1 have n=0; wherein from about 0.001% to about 25% by weight of the first surfactant are surfactants of Formula 2; and wherein X is a hydrophilic moiety; b) from about 0.1% to about 5% by weight of the composition of an encapsulate comprising a shell and a core,

2

wherein the shell comprises a polyacrylate and the core comprises a perfume; and c) a detergent adjunct.

Also included herein, for example, is a liquid detergent composition, comprising a) a first surfactant consisting essentially of a mixture of surfactant isomers of Formula 1 and surfactants of Formula 2:

Formula 2:
$$CH_3$$
— $(CH_2)_{m+n+3}$ — X

wherein from about 50% to about 100% by weight of the first surfactant are isomers having m+n=11; wherein between about 25% to about 50% of the mixture of surfactant isomers of Formula 1 have n=0; wherein from about 0.001% to about 25% by weight of the first surfactant are surfactants of Formula 2; and wherein X is a hydrophilic moiety; and b) an encapsulate comprising a shell and a core, wherein the shell comprises a polyacrylate and the core comprises a perfume; wherein the ratio by weight of the first surfactant to the encapsulate is from about 300:1 to about 2:1.

These and other incarnations will be more fully described throughout the specification.

DETAILED DESCRIPTION OF THE INVENTION

Consumers can equate the scent of a substrate to its level of cleanliness. In addition, some consumers generally prefer a strongly scented substrate after a wash. While this can at least partially be achieved with the use of perfume, depositing perfume onto a substrate can be inefficient meaning much of the perfume will go down the drain. In addition, perfume can be expensive, so adding more perfume to a product adds to the cost of a product. Moreover, formulating with high levels of perfume can lead to issues with the stability of the product.

This has led to the development of perfume technologies. One such perfume technology is an encapsulate. Generally speaking, an encapsulate has a shell and a core. The shell is triggered by an event (like moisture or friction) and releases the contents stored in the core, which can be, for example, a perfume. Encapsulates, however, can have their own difficulties. For example, like perfume, they can be difficult to deposit onto a target substrate. As such, the present inventors investigated whether it is possible to find synergies between certain surfactants and encapsulates which could help with the deposition of the encapsulate onto a target substrate.

5

10

15

20

25

One surfactant investigated was an anionic surfactant comprising a branched alkyl sulfate (a mixture of surfactant isomers of Formula 1 and surfactants of Formula 2:

CH₂—X
I
Formula 1: CH₃—(CH₂)_m—CH—(CH₂)_n—CH₃

$$6 \le m \le 11$$
; $0 \le n \le 5$;

Formula 2: CH_3 — $(CH_2)_{m+n+3}$ —X

wherein from about 50% to about 100% by weight of the first surfactant are isomers having m+n = 11; wherein between about 25% to about 50% of the mixture of surfactant isomers of Formula 1 have n = 0; wherein from about 0.001% to about 25% by weight of the first surfactant are surfactants of Formula 2; and wherein X is a hydrophilic moiety). The encapsulate investigated was a polyacrylate shell with perfume as the core.

To investigate whether a synergy exists between these materials, a liquid detergent composition is made (Comparative Composition A). This composition is a liquid detergent chassis without either the branched alkyl sulfate or the encapsulate. Comparative Compositions B, C, and D are also made which is the liquid detergent chassis with the encapsulate and Comparative Compositions E, F, and G are the liquid detergent chassis with the addition of the branched alkyl sulfate. Inventive Compositions 1-5 are made with both the branched alkyl sulfate and the encapsulate. The formulas for Comparative Compositions A-G and Inventive Compositions 1-5 are in the Examples section below.

The encapsulate deposition of a target substrate is measured utilizing a fabric headspace analysis. Typically, greater perfume intensity (as measured by headspace analysis) correlates with higher concentrations of encapsulates on the target substrate. Here terry fabrics are obtained from Calderon (Indianapolis, IN, USA). The terry fabrics are stripped, pre-conditioned, and then run through wash tests. The specifics for this process can be found in the Methods section called Fabric Headspace Analysis Method.

When looking for synergy, one is looking for more than an additive effect. So, one looks at the impact of each of the given materials individually, the expected effect of utilizing them together, and the actual effect of using them together. Headspace intensity is calculated utilizing a single point calibration of the perfume raw materials. The total headspace concentration for each vial is calculated from the sum of the concentration of each detected perfume raw material, and the headspace is averaged for the treated fabrics. In addition, to take into account the chassis (Comparative Composition A) and any benefits seen from the chassis, the values in Tables 1-2 determined relative to Comparative Composition A (Delta Headspace vs Comparative Comp. A).

5

10

15

20

25

4

For Table 1, the dry fabric odor is measured, as discussed in further detail in the Fabric Headspace Analysis Method. As can be seen in Table 1 below, the actual headspace level of Inventive Composition 1 is 0.15 nmol/L more, or 26% more, than what we be expected based on the individual measurements of Comparative Compositions B and E. The same is also true for Inventive Compositions 2-5 whose actual headspace values are between 22% and 52% more than what would be expected based on the performance of the individual components in the chassis. This indicates a synergy between the branched alkyl sulfate and the encapsulate which results in improved deposition of the encapsulate onto the target substrate, particularly for terry cloth.

TABLE 1

| | Comparative Comp. (B) | Comparative Comp. (E) | Expected Result (B) + (E) | Actual of Inventive Comp. (1) | Synergy (Actual Inv. Comp. 1 > B + E) |
|--|--------------------------|--------------------------|---------------------------------|----------------------------------|--|
| Delta Headspace vs Comparative Comp. (A) | 0.57 nmol/L | 0.01 nmol/L | 0.58 nmol/L | 0.73 nmol/L | 26% more than expected |
| | | | | | |
| | Comparative Comp. (C) | Comparative Comp. (E) | Expected Result (C) + (E) | Actual of Inventive Comp. (2) | Synergy (Actual Inv. Comp. 2 > C + E) |
| Delta Headspace vs Comparative Comp. (A) | 1.32 nmol/L | 0.01 nmol/L | 1.33 nmol/L | 1.62 nmol/L | 22% more than expected |
| | | | | | |
| | Comparative Comp. (C) | Comparative Comp. (F) | Expected Result (C) + (F) | Actual of Inventive Comp (3) | Synergy (Actual Inv. Comp. 3 > C + F) |
| Delta Headspace vs Comparative Comp. (A) | 1.32 nmol/L | -0.02 nmol/L | 1.30 nmol/L | 1.94 nmol/L | 49% more than expected |
| | | | | | |
| | Comparative Comp. (C) | Comparative Comp. (G) | Expected Result (C) + (G) | Actual of Inventive Comp (4) | Synergy (Actual Inv. Comp. 4 > C + G) |
| Delta Headspace vs Comparative Comp. (A) | 1.32 nmol/L | -0.02 nmol/L | 1.30 nmol/L | 1.98 nmol/L | 52% more than expected |
| | | | | | |
| | Comparative Comp. (D) | Comparative Comp. (G) | Expected Result (D) + (G) | Actual of Inventive Comp (5) | Synergy (Actual Inv. Comp. 5 > D + G) |
| Delta Headspace vs Comparative Comp. (A) | 3.66 nmol/L | -0.02 nmol/L | 3.64 nmol/L | 5.08 nmol/L | 40% more than expected |

Additional testing is completed with crushed fabric samples. The crushed fabric samples are prepared the same as the dry fabric samples with the exception that crushed samples are uncapped at the apparatus and a rod is lowered into the vial which applies 67psi of equal weight for 10 seconds to the fabric in the vial. As can be seen in Table 2 below, the synergy is even more pronounced when looking at the crushed fabric samples.

TABLE 2

| | Comparative Comp. (B) | Comparative Comp. (E) | Expected Result (B) + (E) | Actual of Inventive Comp. (1) | Synergy (Actual Inv. Comp. 1 > B + E) |
|--|-----------------------|-----------------------|---------------------------------|----------------------------------|--|
| Delta Headspace vs Comparative Comp. (A) | 0.83 nmol/L | 0.03 nmol/L | 0.86 nmol/L | 1.19 nmol/L | 38% more than expected |
| | | | | | |
| | Comparative Comp. (C) | Comparative Comp. (E) | Expected Result (C) + (E) | Actual of Inventive Comp. (2) | Synergy (Actual Inv. Comp. 2 > C + E) |
| Delta Headspace vs Comparative Comp. (A) | 1.94 nmol/L | 0.03 nmol/L | 1.97 nmol/L | 2.65 nmol/L | 35% more than expected |
| | | | | | |
| | Comparative Comp. (C) | Comparative Comp. (F) | Expected Result (C) + (F) | Actual of Inventive Comp (3) | Synergy (Actual Inv. Comp. 3 > C + F) |
| Delta Headspace vs Comparative Comp. (A) | 1.94 nmol/L | -0.02 nmol/L | 1.92 nmol/L | 3.07 nmol/L | 60% more than expected |
| | | | | | |
| | Comparative Comp. (C) | Comparative Comp. (G) | Expected Result (C) + (G) | Actual of Inventive Comp (4) | Synergy (Actual Inv. Comp. 4 > C + G) |
| Delta Headspace vs Comparative Comp. (A) | 1.94 nmol/L | -0.03 nmol/L | 1.91 nmol/L | 3.52 nmol/L | 84% more than expected |
| | | | | | |
| | Comparative Comp. (D) | Comparative Comp. (G) | Expected Result (D) + (G) | Actual of Inventive Comp (5) | Synergy (Actual Inv. Comp. 5 > D + G) |
| Dolta Headspace vs Comparative Comp. (A) | 5.51 nmol/L | -0.03 nmol/L | 5.48 nmol/L | 8.10 nmol/L | 48% more than expected |

Given the synergy observed between the branched alkyl sulfate and the encapsulate, it is believed a liquid detergent formulation can be formulated which can have less perfume encapsulates, but have a similar or better aroma performance to a liquid detergent with a higher level of encapsulates without a branched alkyl sulfate. This can give additional formulation flexibility, cost savings, and provide opportunities for a more sustainable formula.

LIQUID DETERGENT COMPOSITION

A liquid detergent composition can include a first surfactant comprising a branched alkyl sulfate and an encapsulate. The liquid detergent composition may comprise from about 5% to about 60% by weight of total surfactant. The liquid detergent composition may comprise from about 5%, 6%, 7%, 8%, 9%, or 10% to about 8%, 9%, 10%, 12%, 14%, 16%, 18%, 20%, 22%, 24%, 26%, 28%, 30%, 32%, 34%, 36%, 38%, 40%, 45%, 50%, or any combination thereof, by weight of the composition of total surfactant. The ratio by weight of the first surfactant to the encapsulate can be from about 300:1 to about 2:1, from about 200:1 to about 2:1, from about 100:1 to about 2:1, from about 50:1 to about 2:1, from about 30:1 to about 2:1. The liquid detergent composition may also comprise from about 1% to about 95% of a carrier, like water. The liquid detergent composition can be a laundry detergent composition. A liquid "laundry detergent composition" includes any composition capable of cleaning fabric in a washing machine or in a hand wash context. The liquid laundry detergent compositions can be used in high efficiency and standard washing machines, in addition to hand washing in a tub or basin for example.

The liquid detergent composition can have a greater dry fabric odor than the combination of dry fabric odor of a first reference composition comprising the first surfactant and a second reference composition comprising the encapsulate. The liquid detergent composition can have a greater crushed fabric odor than the combination of crushed fabric odor of a first reference composition comprising the first surfactant and a second reference composition comprising the encapsulate. The dry fabric odor and/or the crushed fabric odor may be about 10% or more, about 29% or more, or about 30% or more above that of the combination of the first reference composition comprising the encapsulate. The first reference composition would not contain the microcapsule and the second reference composition would not contain the first surfactant. An example of a chassis which can be used to make the first and second reference compositions is Comparative Example A. The dry fabric and crushed fabric odor values can be measured as disclosed herein. The fabric used for the measurement can be, for example, on a terry fabric.

5

10

15

20

25

Branched Alkyl Sulfate

5

10

15

20

25

A liquid detergent composition can comprise from about 1% to about 30% by weight of the composition of a first surfactant comprising a branched alkyl sulfate. The liquid detergent composition may also comprise from about 1%, 2%, 3%, 4%, 5%, 6%, 7%, 8%, 9%, or 10% to about 5%, 6%, 7%, 8%, 9%, 10%, 12%, 14%, 16%, 18%, 20%, 22%, 24%, 26%, 28%, or any combination thereof, by weight of the composition of a branched alkyl sulfate. The branched alkyl sulfate can comprise a 2-alkyl branched alkyl alcohol. 2-alkyl branched alcohols are positional isomers, where the location of the hydroxymethyl group (consisting of a methylene bridge (-CH₂- unit) connected to a hydroxy (-OH) group) on the carbon chain varies. Thus, a 2alkyl branched alkyl alcohol is generally composed of a mixture of positional isomers. Furthermore, it is well known that fatty alcohols, such as 2-alkyl branched alcohols, and surfactants are characterized by chain length distributions. In other words, fatty alcohols and surfactants are generally made up of a blend of molecules having different alkyl chain lengths (though it is possible to obtain single chain-length cuts). Notably, the 2-alkyl primary alcohols described herein, which may have specific alkyl chain length distributions and/or specific fractions of certain positional isomers, cannot be obtained by simply blending commercially available materials. Specifically, the distribution of from about 50% to about 100% by weight surfactants having m+n = 11 is not achievable by blending commercially available materials.

The liquid detergent composition can comprise a first surfactant, wherein said first surfactant consists essentially of a mixture of surfactant isomers of Formula 1 and surfactants of Formula 2:

$$CH_2 - X$$
I
(1) $CH_3 - (CH_2)_m - CH - (CH_2)_n - CH_3$ $6 \le m \le 11$; $0 \le n \le 5$;

(2)
$$CH_3 - (CH_2)_{m+n+3} - X$$

wherein from about 50% to about 100% by weight of the first surfactant are isomers having m+n = 11; wherein from about 25% to about 50% of the mixture of surfactant isomers of Formula 1 have n=0; wherein from about 0.001% to about 25% by weight of the first surfactant are surfactants of Formula 2; and wherein X is a hydrophilic moiety.

X can be, for example, neutralized with sodium hydroxide, potassium hydroxide, magnesium hydroxide, lithium hydroxide, calcium hydroxide, ammonium hydroxide, monoethanolamine,

diethanolamine, triethanolamine, monoisopropanolamine, diamine, polyamine, primary amine, secondary amine, tertiary amine, amine containing surfactant, or a combination thereof.

X may be selected from sulfates, alkoxylated alkyl sulfates, sulfonates, amine oxides, polyalkoxylates, polyhydroxy moieties, phosphate esters, glycerol sulfonates, polygluconates, polyphosphate esters, phosphonates, sulfosuccinates, sulfosuccaminates, polyalkoxylated carboxylates, glucamides, taurinates, sarcosinates, glycinates, isethionates, dialkanolamides, monoalkanolamides, monoalkanolamide sulfates, diglycolamides, diglycolamide sulfates, glycerol esters, glycerol esters sulfates, glycerol ethers, glycerol ether sulfates, polyglycerol ethers, polyglycerol ether sulfates, sorbitan esters, polyalkoxylated sorbitan esters, ammonioalkanesulfonates, amidopropyl betaines, alkylated quats, alkylated/polyhydroxylated oxypropyl quats, imidazolines, 2-yl-succinates, sulfonated alkyl esters, sulfonated fatty acids, and mixtures thereof.

The first surfactant may have between about 15% to about 40% of the mixture of surfactant isomers of Formula 1 have n=1, such as, for example between about 20% to about 40%, between about 25% to about 35%, or between about 30% to about 40%. The first surfactant may have between about 60% to about 90% of the mixture of surfactant isomers of Formula 1 have n<3, such as, for example between about 65% and 85%, between about 70% and 90%, or between about 80% and 90%. The detergent composition may have between about 90% to about 100% of the first surfactant where the isomers have m+n=11, such as, for example between about 95% and 100%.

The first surfactant may have from about 15% to about 40% by weight of the first surfactant mixture are isomers of Formula 1 with n=1 and from about 5% to about 20% by weight of the first surfactant mixture are isomers of Formula 1 with n=2. The first surfactant may have no isomers of Formula 1 with n equal to or greater than 6. The first surfactant may have up to about 40% of the mixture of surfactant isomers of Formula 1 with n > 2. The first surfactant may have up to about 25% of the mixture of surfactant isomers of Formula 1 have n > 2. The first surfactant may have up to about 20% by weight of the Formula 2 isomer.

Impurities

The process of making the 2-alkyl primary alcohol-derived surfactants described above may produce various impurities and/or contaminants at different steps of the process.

The C14 olefin and C12 olefin sources used in the hydroformylation to make the starting C15 aldehydes and C13 aldehydes and subsequent alcohols and corresponding surfactants of use in the present invention may have low levels of impurities that lead to impurities in the starting

5

10

15

20

25

C15 alcohols and C13 alcohol and therefore also in the C15 alkyl sulfate and C13 alkyl sulfate. While not intending to be limited by theory, such impurities present in the C14 olefin and C12 olefin feeds can include vinylidene olefins, branched olefins, paraffins, aromatic components, and low levels of olefins having chain-lengths other than the intended 14 carbons or 12 carbons. Branched and vinylidene olefins are typically at or below 5% in C14 and C12 alpha olefin sources. Impurities in the resulting C15 alcohols and C13 alcohols can include low levels of linear and branched alcohols in the range of C10 to C17 alcohols, especially C11 and C15 alcohols in the C13 alcohol, and especially C13 and C17 alcohols in the C15 alcohol, typically less than 5% by weight of the mixture, preferably less than 1%; low levels of branching in positions other than the 2-alkyl position resulting from branched and vinylidene olefins are typically less than about 5% by weight of the alcohol mixture, preferably less than 2%; paraffins and olefins, typically less than 1% by weight of the alcohol mixture, preferably less than about 0.5%; low levels of aldehydes with a carbonyl value typically below 500 mg/kg, preferably less than about 200 mg/kg. These impurities in the alcohol can result in low levels of paraffin, linear and branched alkyl sulfates having total carbon numbers other than C15 or C13, and alkyl sulfates with branching in positions other than the 2-alkyl location, wherein these branches can vary in length, but are typically linear alkyl chains having from 1 to 6 carbons. The step of hydroformylation may also yield impurities, such as linear and branched paraffins, residual olefin from incomplete hydroformylation, as well as esters, formates, and heavy-ends (dimers, trimers). Impurities that are not reduced to alcohol in the hydrogenation step may be removed during the final purification of the alcohol by distillation.

Also, it is well known that the process of sulfating fatty alcohols to yield alkyl sulfate surfactants also yields various impurities. The exact nature of these impurities depends on the conditions of sulfation and neutralization. Generally, however, the impurities of the sulfation process include one or more inorganic salts, unreacted fatty alcohol, and olefins ("The Effect of Reaction By-Products on the Viscosities of Sodium Lauryl Sulfate Solutions," <u>Journal of the American Oil Chemists' Society</u>, Vol. 55, No. 12, p. 909-913 (1978), C.F. Putnik and S.E. McGuire).

Alkoxylation impurities may include dialkyl ethers, polyalkylene glycol dialkyl ethers, olefins, and polyalkylene glycols. Impurities can also include the catalysts or components of the catalysts that are used in various steps.

Encapsulates

5

10

15

20

25

A liquid detergent compositions can comprise an encapsulate. The composition may comprise, for example, from about 0.05% to about 5%, or from about 0.05% to about 5%, or from about 0.1% to about 5%, or from about 0.2% to about 2%, by weight of the composition, of encapsulates. The composition may comprise a sufficient amount of encapsulates to provide from about 0.05% to about 10%, or from about 0.1% to about 5%, or from about 0.1% to about 2%, by weight of the composition, of perfume to the composition. The encapsulate comprises a shell and a core. The core may be surrounded by the shell.

When discussing herein the amount or weight percentage of the encapsulates, it is meant the sum of the shell material and the core material.

The encapsulates may have a volume weighted median encapsulate size from about 0.5 microns to about 100 microns, or even 10 to 100 microns, preferably from about 1 micron to about 60 microns, or even 10 microns to 50 microns, or even 20 microns to 45 microns, or alternatively 20 microns to 60 microns.

Core

5

10

15

20

25

The core may comprise a perfume. The perfume may comprise a single perfume raw material or a mixture of perfume raw materials. The term "perfume raw material" (or "PRM") as used herein refers to compounds having a molecular weight of at least about 100 g/mol and which are useful in imparting an odor, fragrance, essence, or scent, either alone or with other perfume raw materials. Typical PRMs comprise inter alia alcohols, ketones, aldehydes, esters, ethers, nitrites and alkenes, such as terpene. A listing of common PRMs can be found in various reference sources, for example, "Perfume and Flavor Chemicals", Vols. I and II; Steffen Arctander Allured Pub. Co. (1994) and "Perfumes: Art, Science and Technology", Miller, P. M. and Lamparsky, D., Blackie Academic and Professional (1994).

The perfume in the core may contain a mixture of perfume raw materials. The perfume in the core may comprise at least three, or at least four, or at least five, or at least six, or at least seven, or at least eight, or at least nine, or at least ten perfume raw materials. A mixture of perfume raw materials may provide more complex and desirable aesthetics, and/or better perfume performance or longevity, for example at a variety of touchpoints.

It may be that the perfume in the core comprises less than about fifty, or less than about forty, or less than about thirty, or less than about twenty-five, or less than about twenty perfume

raw materials. It may be desirable to limit the number of perfume raw materials in the perfume as a way to reduce or limit formulation complexity and/or cost.

The perfume may comprise at least one perfume raw material that is naturally derived. Such components may be desirable for sustainability/environmental reasons. Naturally derived perfume raw materials may include natural extracts or essences, which may contain a mixture of PRMs. Such natural extracts or essences may include orange oil, lemon oil, rose extract, lavender, musk, patchouli, balsamic essence, sandalwood oil, pine oil, cedar, and the like.

The core of the encapsulates of the present disclosure may comprise a partitioning modifier. The core may comprise, in addition to the encapsulated benefit agent, from greater than 0% to about 80%, preferably from greater than 0% to about 50%, more preferably from greater than 0% to about 30%, most preferably from greater than 0% to about 20%, based on total core weight, of a partitioning modifier.

The partitioning modifier may comprise a material selected from the group consisting of vegetable oil, modified vegetable oil, mono-, di-, and tri-esters of C₄-C₂₄ fatty acids, isopropyl myristate, dodecanophenone, lauryl laurate, methyl behenate, methyl laurate, methyl palmitate, methyl stearate, and mixtures thereof. The partitioning modifier may preferably comprise or consist of isopropyl myristate. The modified vegetable oil may be esterified and/or brominated. The modified vegetable oil may preferably comprise castor oil and/or soybean oil. US Patent Application Publication 20110268802, incorporated herein by reference, describes other partitioning modifiers that may be useful in the presently described perfume encapsulates.

Shell

The encapsulates may comprise a shell. The shell may, partially or completely, surround the core. The wall material may include an aminoplast. The aminoplast may include a polyurea, polyurethane, and/or polyurea urethane. The aminoplast may include an aminoplast copolymer, such as melamine-formaldehyde, urea-formaldehyde, cross-linked melamine formaldehyde, or mixtures thereof. The wall may include melamine formaldehyde, which may further include a coating as described below. The encapsulate may include a core that comprises perfume, and a wall that includes melamine formaldehyde and/or cross linked melamine formaldehyde. The encapsulate may include a core that comprises perfume, and a wall that comprises melamine formaldehyde and/or cross linked melamine formaldehyde, poly(acrylic acid) and poly(acrylic acid-co-butyl acrylate).

30

5

10

15

20

The shell may comprise a polymeric material. The polymeric material may comprise a (meth)acrylate material. As described above, perfumes having an acid value of greater than 5.0 mg KOH/g have been found to perform surprising well when encapsulated in a shell comprising an acrylate material. The polymeric material of the shell may be formed, at least in part, by a radical polymerization process.

The acrylate material of the shell may include a (meth)acrylate material selected from the group consisting of a polyacrylate, a polyethylene glycol acrylate, a polyurethane acrylate, an epoxy acrylate, a polymethacrylate, a polyethylene glycol methacrylate, a polyurethane methacrylate, an epoxy methacrylate, and mixtures thereof.

As used herein, reference to the term "(meth)acrylate" or "(meth)acrylic" is to be understood as referring to both the acrylate and the methacrylate versions of the specified monomer, oligomer and/or prepolymer. For example, "allyl (meth)acrylate" indicates that both allyl methacrylate and allyl acrylate are possible, similarly reference to alkyl esters of (meth)acrylic acid indicates that both alkyl esters of acrylic acid and alkyl esters of methacrylic acid are possible, similarly poly(meth)acrylate indicates that both polyacrylate and polymethacrylate are possible. Poly(meth)acrylate materials are intended to encompass a broad spectrum of polymeric materials including, for example, polyester poly(meth)acrylates, urethane and polyurethane poly(meth)acrylates (especially those prepared by the reaction of an hydroxyalkyl (meth)acrylate with a polyisocyanate or a urethane polyisocyanate), methylcyanoacrylate, ethylcyanoacrylate, diethyleneglycol di(meth)acrylate, trimethylolpropane tri(meth)acrylate, ethylene glycol di(meth)acrylate, allyl (meth)acrylate, glycidyl (meth)acrylate, (meth)acrylate functional silicones, di-, tri- and tetraethylene glycol di(meth)acrylate, dipropylene glycol di(meth)acrylate, polyethylene glycol di(meth)acrylate, di(pentamethylene glycol) di(meth)acrylate, ethylene di(meth)acrylate, neopentyl glycol di(meth)acrylate, trimethylol propane tri(meth)acrylate, ethoxylated bisphenol A di(meth)acrylates, bisphenol A di(meth)acrylates, diglycerol di(meth)acrylate, tetraethylene glycol dichloroacrylate, 1,3butanediol di(meth)acrylate, neopentyl di(meth)acrylate, trimethylolpropane tri(meth)acrylate, polyethylene glycol di(meth)acrylate and dipropylene glycol di(meth)acrylate and various multifunctional (meth) acrylates. Monofunctional acrylates, i.e., those containing only one acrylate group, may also be advantageously used. Typical monoacrylates include 2-ethylhexyl (meth)acrylate, 2-hydroxyethyl (meth)acrylate, cyanoethyl (meth)acrylate, 2-hydroxypropyl (meth)acrylate, p-dimethylaminoethyl (meth)acrylate, lauryl (meth)acrylate, cyclohexyl

30

5

10

15

20

(meth)acrylate, tetrahydrofurfuryl (meth)acrylate, chlorobenzyl (meth)acrylate, aminoalkyl(meth)acrylate, various alkyl(meth)acrylates and glycidyl (meth)acrylate. Mixtures of (meth)acrylates or their derivatives as well as combinations of one or more (meth)acrylate monomers, oligomers and/or prepolymers or their derivatives with other copolymerizable monomers, including acrylonitriles and methacrylonitriles may be used as well.

The main said shell material may comprise polyacrylate. The shell material may include from about 25% to about 100%, or from about 50% to about 100%, or from about 65% to about 100%, by weight of the shell material, of a polyacrylate polymer. The polyacrylate may include a polyacrylate cross linked polymer.

The (meth)acrylate material of the encapsulates may include a polymer derived from a material that comprises one or more multifunctional acrylate moieties. The multifunctional acrylate moiety may be selected from the group consisting of tri-functional acrylate, tetra-functional acrylate, penta-functional acrylate, hexa-functional acrylate, hepta-functional acrylate and mixtures thereof. The multifunctional acrylate moiety is preferably hexa-functional acrylate. The acrylate material may include a polyacrylate that comprises a moiety selected from the group consisting of an acrylate moiety, methacrylate moiety, amine acrylate moiety, amine methacrylate moiety, a carboxylic acid acrylate moiety, and combinations thereof, preferably an amine methacrylate or carboxylic acid acrylate moiety.

The (meth)acrylate material may include a material that comprises one or more multifunctional acrylate and/or multifunctional methacrylate moieties. The ratio of material that comprises one or more multifunctional acrylate moieties to material that comprises one or more methacrylate moieties may be from about 999:1 to about 6:4, preferably from about 99:1 to about 8:1, more preferably from about 99:1 to about 8.5:1.

Examples of multifunctional acrylates include commercial materials from Sartomer Inc., such as CN975 (a hexafunctional aromatic urethane acrylate), CN9006 (a hexafunctional aliphatic urethane acrylate), CN296, CN293, CN295 (a hexafunctional polyester acrylate oligomer or acrylated polyester), CN2282, CN294E, CN299 (a tetrafunctional polyester acrylate oligomer or acrylated polyester), SR494, SR295, SR255 (a tetrafunctional acrylate oligomer), SR9009, SR9011 (a trifunctional methacrylate oligomer), SR929 (a polyester urethane acrylate oligomer), SR9053 (an acid ester trifunctional acrylate oligomer), CN989, CN9301 (an aliphatic

30

5

10

15

20

urethane acrylate), SR350, SR353 (a trifunctional acrylate oligomer), SR9012 (a trifunctional acrylate ester), and/or SR368 (a tris (2-hydroxyethyl)isocyanurate triacrylate).

The acrylate material may be derived from a monomer selected from a hexafunctional acrylate, a triacrylate, or mixtures thereof, preferably a hexafunctional aromatic acrylate, an isocyanurate triacrylate, or mixtures thereof, more preferably a hexafunctional aromatic urethane acrylate, a tris (2-hydroxyethyl)isocyanurate triacrylate, or mixtures thereof, as such materials have be found to be useful in making robust capsules.

The shells of the particles described herein may comprise a poly(meth)acrylate polymer comprising a reaction product of at least one monomer or oligomer thereof. The monomer comprises a structure according to Formula I:

$$\begin{bmatrix} R^2 \\ I \\ R^1 \text{- O-C - C=CH}_2 \end{bmatrix}_{n}$$

wherein R^1 is selected from C_1 to C_8 , R^2 is hydrogen or methyl, wherein n is an integer from 1 to 3, and A is a ring structure selected from any of those selected from Formulas II-VI:

The shell of the encapsulates may be substantially free of melamine derivatives. Melamine derivatives may include polymers or other material that are derived from melamine-

15

5

based monomers, for example melamine-formaldehyde material. The shell of the encapsulates may be substantially free of melamine-formaldehyde material. Without wishing to be bound by theory, it is believed that melamine-formaldehyde materials provide relatively strong negative charges to encapsulate shells, resulting in poor interactions and/or performance with quaternary ammonium ester compounds, particular certain compounds comprising triester quats and/or derived from triethanolamines.

The encapsulate, based on total encapsulate weight, may comprise from about 0.5% to about 40%, more preferably 0.8% to 5% of an emulsifier. Emulsifiers may be useful as processing aids during formation of the encapsulates. The emulsifier may be embedded in and/or located on the shell. The emulsifier may be selected from the group consisting of polyvinyl alcohol, carboxylated or partially hydrolyzed polyvinyl alcohol, methyl cellulose, hydroxyethylcellulose, carboxymethylcellulose, methylhydroxypropylcellulose, salts or esters of stearic acid, lecithin, organosulphonic acid, 2-acrylamido-2-alkylsulphonic acid, styrene sulphonic acid, polyvinylpyrrolidone, copolymers of N-vinylpyrrolidone, polyacrylic acid, polymethacrylic acid; copolymers of acrylic acid and methacrylic acid, and water-soluble surfactant polymers which lower the surface tension of water.

The emulsifier preferably comprises polyvinyl alcohol. Preferably, the polyvinylalcohol has at least one the following properties, or a mixture thereof: (i) a hydrolysis degree from 70% to 99%, preferably 75% to 98%, more preferably from 80% to 96%, more preferably from 82% to 96%, most preferably from 86% to 94%; and/or (ii) a viscosity of from 2 mPa.s to 150 mPa.s, preferably from 3 mPa.s to 70 mPa.s, more preferably from 4 mPa.s to 60 mPa.s, even more preferably from 5 mPa.s to 55 mPa.s in 4% water solution at 20°C. Suitable polyvinylalcohol materials may be selected from Selvol 540 PVA (Sekisui Specialty Chemicals, Dallas, TX), Mowiol 18-88 = Poval 18-88, Mowiol 3-83, Mowiol 4-98 = Poval 4-98 (Kuraray), Poval KL-506 = Poval 6-77 KL (Kuraray), Poval R-1130 = Poval 25-98 R (Kuraray), Gohsenx K-434 (Nippon Gohsei).

The encapsulates of the present disclosure may comprise a coating. The shell may comprise the coating; for example, the coating may be on an outer surface of the shell. The encapsulates may be manufactured and be subsequently coated with a coating material. The coating may be useful as a deposition aid. Non-limiting examples of coating materials include but are not limited to materials selected from the group consisting of poly(meth)acrylate, poly(ethylene-maleic anhydride), polyamine, wax, polyvinylpyrrolidone, polyvinylpyrrolidone

30

5

10

15

20

co-polymers, polyvinylpyrrolidone-ethyl acrylate, polyvinylpyrrolidone- vinyl acrylate. polyvinylpyrrolidone methacrylate, polyvinylpyrrolidone/vinyl acetate, polyvinyl acetal, polyvinyl butyral, polysiloxane, poly(propylene maleic anhydride), maleic anhydride derivatives, co-polymers of maleic anhydride derivatives, polyvinyl alcohol, styrene-butadiene latex, gelatin, gum Arabic, carboxymethyl cellulose, carboxymethyl hydroxyethyl cellulose, hydroxyethyl cellulose, other modified celluloses, sodium alginate, chitosan, casein, pectin, modified starch, polyvinyl acetal, polyvinyl butyral, polyvinyl methyl ether/maleic anhydride, polyvinyl pyrrolidone and its co polymers, poly(vinyl pyrrolidone/methacrylamidopropyl trimethyl ammonium chloride), polyvinylpyrrolidone/vinyl acetate, polyvinyl pyrrolidone/dimethylaminoethyl methacrylate, polyvinyl amines, polyvinyl formamides, polyallyl amines and copolymers of polyvinyl amines, polyvinyl formamides, and polyallyl amines and mixtures thereof. The coating material may be a cationic polymer. The coating material may comprise chitosan.

The compositions may comprise encapsulates according to the present disclosure wherein at least 75% of the encapsulates have an encapsulate shell thickness of from about 10 nm to about 350 nm, from about 20 nm to about 200 nm, or from 25 nm to about 180 nm, as determined by the Encapsulate Shell Thickness test method described herein.

Additional Surfactants

The liquid detergent composition may further comprise an additional surfactant. The additional surfactant may be present at a level of about 0.25% to about 50% by weight of the liquid detergent composition. The additional surfactant may be anionic, nonionic, cationic, zwitterionic, amphoteric, or a combination thereof. The additional surfactant may comprise an alkyl sulfate, an alkyl ethoxy sulfate (AES), alkyl benzene sulfonate, ethoxylated alcohol nonionic surfactant, amine oxide, methyl ester sulfonate, glycolipid surfactant, alkylpolyglucoside surfactant, or combinations thereof. The additional surfactant may be selected from the group consisting of an alkyl ethoxy sulfate, alkyl benzene sulfonate, an ethoxylated alcohol nonionic surfactant, an amine oxide surfactant, and mixtures thereof. The additional surfactant may comprise a linear alkyl benzene sulfonate, alkyl sulfate, alkyl ethoxylated sulfate, a nonionic surfactant, or a combination thereof.

A liquid detergent composition can comprise an additional surfactant comprising AES. The liquid detergent composition may also comprise from about 1%, 2%, 3%, 4%, 5%, 6%, 7%, 8%, 9%, or 10% to about 5%, 6%, 7%, 8%, 9%, 10%, 12%, 14%, 16%, 18%, 20%, 22%, 24%, 26%, 28%, or any combination thereof, by weight of the composition of an alkyl ethoxy sulfate.

30

5

10

15

20

The AES surfactant comprises a plurality of AES compounds, where each AES compound comprises an alkyl chain. The alkyl chain of a particular AES compound may be characterized by the total number of carbons in the alkyl portion, otherwise known as the alkyl chain lengths. A given amount of AES surfactant may include a variety of AES compounds having chain lengths that fall within certain proportions or distributions. Thus, a given amount or sample of AES may be characterized by distributions of AES compounds having certain chain lengths, and/or by a weight average number of carbons in the alkyl portion.

Commercially available AES surfactants may include AES having weight average chain lengths of from twelve to fifteen, known as C12-15 AES, or chain lengths of from twelve to fourteen, known as C12-14 AES. While these are described as weight average chain lengths of a certain range, there could be materials within these which have carbon chain lengths outside of the specified range. As long as the weight average chain length of an AES material falls within the ranges as defined, the material is included in the scope even if there are some carbon chain lengths outside of the specified length.

Another AES surfactant suitable for use herein may include a relatively high proportion of an AES compound having fifteen carbon atoms in the alkyl chain ("C15 AES"). C15 AES may be desirable because the relatively longer alkyl chain increases the hydrophobicity of the AES surfactant, which may provide improved soil removal, such as greasy soil removal. The AES surfactant may include from about 40wt%, or from about 45wt%, to about 70wt%, or to about 60wt%, by weight of the AES surfactant, of C15 AES. C15 AES may make up a major portion of the AES surfactant, meaning that there is more C15 AES surfactant by weight present than any other single type of AES surfactant. C15 AES may make up at least half, or even a majority, of the AES surfactant by weight.

The AES surfactant may include an AES compound having fourteen carbon atoms in the alkyl chain ("C14 AES"), for example at least about 1wt%, by weight of the AES surfactant, of C14 AES. The AES surfactant may include relatively limited amounts of C14 AES. For example, the AES surfactant may contain no more than about 30wt%, or no more than about 25wt%, or no more than about 20wt%, or no more than about 15wt%, or no more than about 10wt%, by weight of the AES surfactant, of C14 AES. When a composition or surfactant system comprises a relatively large proportion of C15 AES, it may be desirable to limit the amount of C14 AES, e.g., for stability reasons.

The AES surfactant may include an AES compound having thirteen carbon atoms in the alkyl chain ("C13 AES"). C13 AES may be desirable because the relatively shorter alkyl chain

5

10

15

20

25

decreases the relative hydrophobicity of the AES surfactant, enabling it to remove different soils and/or be relatively more physically stable than a more hydrophobic AES surfactant. The AES surfactant may include from about 15wt%, or from about 20wt%, or from about 25wt%, to about 50wt%, or to about 40wt%, or to about 35wt%, by weight of the AES surfactant, of C13 AES, preferably from about 15wt% to about 35wt%. C13 AES may be present as the first- or second-most prevalent AES compound in the AES surfactant; for example, the AES surfactant may be richest in C15 AES and C13 AES, having relatively high levels of both compared to AES of other chain lengths.

The AES surfactant may include an AES compound having twelve carbon atoms in the alkyl chain ("C12 AES"). The AES surfactant may contain at least about 1wt%, or at least about 3wt%, or at least about 5wt%, or at least about 10wt% of C12 AES. The AES surfactant may contain no more than about 20wt%, or no more than about 15wt%, or no more than about 12wt%, or no more than about 10wt%, or no more than about 5wt%, of C12 AES. The AES surfactant may contain from about 1wt%, or from about 3wt%, to about 20wt%, or to about 15wt%, by weight of the AES surfactant, of C12 AES, preferably from about 3wt% to about 15wt%. C12 AES may be desirable, for example, to counterbalance the hydrophobicity of the C15 AES, resulting in a broader cleaning profile and/or a better stability profile.

The AES surfactant may include at least 1wt%, by weight of the AES surfactant, of each of C12 AES, C13 AES, and C14 AES surfactant, in addition to the amounts of C15 surfactant recited above. The AES surfactant of the present disclosure may comprise from about 30wt% to about 60wt%, by weight of the AES surfactant, of C12 AES, C13 AES, C14 AES, or mixtures thereof, preferably mixtures thereof.

The AES surfactant may comprise from about 1wt% to about 20wt% C12 AES, from about 25wt% to about 50wt% C13 AES, from about 1wt% to about 10wt% C14 AES, and from about 45wt% to about 60wt% C15 AES, wherein each wt% is by weight of the AES surfactant, and may be characterized by alkyl chain lengths having an average molecular weight of from about 205 to about 220, preferably from about 208 to about 218; the provided wt%'s may add up to from about 95wt% to about 100wt%.

The AES surfactant may include an AES compound having sixteen carbon atoms in the alkyl chain ("C16 AES"). The amounts of C16 present may be limited, for example, because the longer chain length may contribute to phase instability. The AES surfactant of the present disclosure may comprise from about 0.1%, by weight of the AES surfactant, to less than about

5

10

15

20

25

5%, or less than about 3%, or less than about 1.5%, or less than 1%, by weight of the AES surfactant, of C16 AES.

The AES surfactant may be characterized by the weight average molecular weight of the chain lengths of the AES compounds in the distribution. The AES surfactant as a whole may be characterized by weight average molecular weight chain lengths that are lower than might be expected in view of the relatively high proportion of C15 AES.

The weight average molecular weight of the chain lengths may be determined by finding the weight average molecular weight of a fatty alcohol consisting of the alkyl chain and a hydroxyl group. Calculating the molecular weight of the chain lengths in such a fashion can present several advantages. For example, AES surfactants are typically synthesized from such fatty alcohols, which serve as a feedstock material before being alkoxylated (e.g., ethoxylated) and sulfated to arrive at the final AES compound(s). Thus, relevant information relating to the fatty alcohol feedstock is typically available from the feedstock supplier and/or the AES manufacturer. Additionally, reporting molecular weight based on a fatty alcohol comprising the alkyl chain rather than the molecular weight of the AES surfactant itself helps to remove uncertainty resulting from variable alkoxylation; for example, a C15 AES material may include some molecules that include one mole of ethoxylation, and others that include two moles and/or three moles of ethoxylation.

For example, the molecular weight of the alkyl chain of a C15 AES compound is based on a C15 fatty alcohol, which may have the following empirical formula: C₁₅H₃₁OH. Such a C15 fatty alcohol has a molecular weight of about 228 daltons. For convenience, Table 4 shows the molecular weight of several exemplary fatty alcohols.

TABLE 4

| Fatty Alcohol, by | Molecular Weight |
|---------------------|------------------|
| carbon chain length | (in daltons) |
| C12 fatty alcohol | 186 |
| C13 fatty alcohol | 200 |
| C14 fatty alcohol | 214 |
| C15 fatty alcohol | 228 |
| C16 fatty alcohol | 242 |

The AES surfactant may be characterized by chain lengths having a weight average molecular weight of from about 200, or from about 205, or from about 208, or from about 210, or

25

5

10

15

from about 211, from about 214, to about 220, or to about 218, or to about 215 daltons, wherein the molecular weight of a particular alkyl chain is based on the molecular weight of fatty alcohol comprising the alkyl chain (i.e., a fatty alcohol consisting of the alkyl chain and a hydroxyl group). The AES surfactant may be characterized by chain lengths having a weight average molecular weight of from about 200 to about 220, or from about 210 to about 220, or from about 211 to about 218 daltons. The AES surfactant may be characterized by chain lengths having a weight average molecular weight of from about 208 to no greater than 215 daltons. AES characterized by chain lengths of a relatively lower weight average molecular weight (e.g., 208-215 daltons) may be particularly preferred in detergent compositions having relatively higher amounts of surfactant (e.g., more than 20wt%), as they facilitate improved physical stability.

AES surfactant may be characterized by their degrees of ethoxylation. In a population of AES compounds, the AES molecules may have varying degrees of ethoxylation. Thus, a given amount or sample of AES may be characterized by a weight average degree of ethoxylation, where the degree of ethoxylation is reported as moles of ethoxy groups (-O-CH₂-CH₂) per mole of AES. The AES surfactant of the present disclosure may be characterized by a weight average degree of ethoxylation of from about 0.5 to about 5, or from about 1 to about 3, or from about 1.5 to about 2.5.

The AES may include at least some alkyl sulfate ("AS") surfactant that is not ethoxylated. The unethoxylated AS may be present as a result of incomplete reactions during the ethoxylation process, and/or because it was added a separate ingredient. For the purposes of the present disclosure, (unethoxylated) AS is considered to be part of the AES surfactant when determining levels, chain length molecular weights, and/or degrees of ethoxylation.

The AES surfactant may comprise AES compounds having linear alkyl chains, AES compounds having branched alkyl chains, or mixtures thereof. The AES surfactant may comprise AES surfactant that is branched at the C2 position, where the C2 is the second carbon away from the ethoxy sulfate head group (i.e., the carbon adjacent the ethoxy sulfate head group is at the C1 position). The AES surfactant may comprise from about 10% to about 30%, by weight of the AES surfactant, of AES surfactant that is branched at the C2 position. Branched alkyl chains may improve and/or broaden the cleaning profile of the AES surfactant. It may also be that linear alkyl portions of the AES compounds are preferred. At least about 50%, or at least about 75%, or at least about 90%, or at least about 95%, or about 100%, by weight of the AES surfactant, of the AES compounds may have alkyl chains that are linear alkyl chains. The AES may comprise a mixture of C15 AES compounds, where at least 60%, by weight of the C15 AES,

5

10

15

20

25

of the C15 AES is linear, and at least 10%, by weight of the C15 AES, of the C15 AES is branched, preferably at the C2 position. The AES may comprise a mixture of C13 AES compounds, where at least 60%, by weight of the C13 AES, of the C13 AES is linear, and at least 10%, by weight of the C13 AES, of the C13 AES is branched, preferably at the C2 position.

As described above, AES compounds are typically manufactured by sulfating an ethoxylated fatty alcohol. A fatty alcohol may first be provided, then ethoxylated according to known methods. Thus, AES compounds, or at least the alkyl chains of the AES compounds, may be described in terms of the sources, for example oils or fatty alcohols, from which they are derived. The AES compounds of the present disclosure may include alkyl chains that are derived from a non-petroleum source, preferably from a natural source. The AES of the present disclosure may include mixtures of AES that includes alkyl chains that are naturally derived and AES that includes alkyl chains of AES that are synthetically derived (e.g., petrol-derived); such mixtures may be useful to account for supply chain variations, disruptions, and/or pricing fluctuations, e.g. so that a shortage of one type of AES may be back-filled by another type.

Natural sources may include oils derived from plants or animal sources, preferably from plants. Representative non-limiting examples of vegetable oils include canola oil, rapeseed oil, coconut oil, corn oil, cottonseed oil, olive oil, palm oil, peanut oil, safflower oil, sesame oil, soybean oil, sunflower oil, linseed oil, palm kernel oil, tung oil, jatropha oil, mustard oil, pennycress oil, camelina oil, castor oil, or mixtures thereof. Suitable feedstock oils may include metathesized oils, typically formed from a metathesis reaction in the presence of a suitable metathesis catalyst. The alkyl portion may be derived from coconut oil, palm kernel oil, or mixtures thereof. Such sources may be desirable for environmental and/or sustainability reasons, as they do not rely on fossil fuels. Further, the alkyl chains of AES compounds derived from natural sources typically contain an even number of carbon atoms.

Other sources of alkyl chains (e.g., feedstock alcohols) may include commercially available alcohols, such as those sold by Shell (e.g., under the NeodolTM tradename, for example NeodolTM 23, NeodolTM 3, NeodolTM 45, and/or NeodolTM 5) and/or Sasol (e.g., LialTM, IsalchemTM, SafolTM, etc.).

It may be that the AES is not derived from a Fischer-Tropsch process. It may be that the AES of the present disclosure is derived from the well-known Shell modified oxo process. The AES of the present disclosure may include AES that is derived from the Ziegler process.

5

10

15

20

25

The AES may be present in acid form, in salt form (e.g., neutralized), or mixtures thereof. The salt-form AES may be an alkali metal salt, preferably a sodium salt, an ammonium salt, or an alkanolamine salt.

The additional surfactant may comprise alkyl benzene sulfonate surfactant. The alkyl group may contain from about 9 to about 15 carbon atoms, in straight chain (linear) or branched chain configuration. The alkyl group may be linear. Such linear alkylbenzene sulfonates are known as "LAS." The linear alkylbenzene sulfonate may have an average number of carbon atoms in the alkyl group of from about 11 to 14. The linear straight chain alkyl benzene sulfonates may have an average number of carbon atoms in the alkyl group of about 11.8 carbon atoms, which may be abbreviated as C11.8 LAS. The linear straight chain alkyl benzene sulfonates may have predominantly 12 carbon atoms in the alkyl group, greater than 50% C12, greater than 75% C12, even greater than 96% C12. The alkyl benzene sulfonate may be present, at least partly, as a salt, such as an alkali metal salt, preferably a sodium salt, or an amine salt, such as an ethanolamine salt, e.g., an monoethanolamine salt.

The additional surfactant may comprise an amine oxide surfactant. Preferred amine oxides are alkyl dimethyl amine oxide or alkyl amido propyl dimethyl amine oxide, more preferably alkyl dimethyl amine oxide and especially coco dimethyl amino oxide. Amine oxide may have a linear or mid-branched alkyl moiety. Typical linear amine oxides include watersoluble amine oxides containing one R1 C8-18 alkyl moiety and 2 R2 and R3 moieties selected from the group consisting of C1-3 alkyl groups and C1-3 hydroxyalkyl groups. Preferably amine oxide is characterized by the formula R1 - N(R2)(R3) O wherein R1 is a C8-18 alkyl and R2 and R3 are selected from the group consisting of methyl, ethyl, propyl, isopropyl, 2-hydroxethyl, 2hydroxypropyl and 3-hydroxypropyl. The linear amine oxide surfactants in particular may include linear C10-C18 alkyl dimethyl amine oxides and linear C8-C12 alkoxy ethyl dihydroxy ethyl amine oxides. Preferred amine oxides include linear C10, linear C10-C12, and linear C12-C14 alkyl dimethyl amine oxides. As used herein "mid-branched" means that the amine oxide has one alkyl moiety having n1 carbon atoms with one alkyl branch on the alkyl moiety having n2 carbon atoms. The alkyl branch is located on the α carbon from the nitrogen on the alkyl moiety. This type of branching for the amine oxide is also known in the art as an internal amine oxide. The compositions of the present disclosure may include from about 0.1% to about 5%, or to about 3%, or to about 1%, by weight of the composition, of amine oxide.

The additional surfactant may comprise a nonionic surfactant. The nonionic surfactant may be an ethoxylated alcohol. The nonionic surfactant may have the formula $R(OC_2H_4)_nOH$,

5

10

15

20

25

wherein R is selected from the group consisting of aliphatic hydrocarbon radicals containing from about 8 to about 16 carbon atoms and the average value of n is from about 5 to about 15. For example, the nonionic surfactant may be selected from ethoxylated alcohols having an average of about 12-14 carbon atoms in the alcohol (alkyl) portion and an average degree of ethoxylation of about 7-9 moles of ethylene oxide per mole of alcohol.

Additional non limiting examples include ethoxylated alkyl phenols of the formula R(OC₂H₄)_nOH, wherein R comprises an alkyl phenyl radicals in which the alkyl groups contain from about 8 to about 12 carbon atoms, and the average value of *n* is from about 5 to about 15, C₁₂-C₁₈ alkyl ethoxylates, such as, NEODOL[®] nonionic surfactants from Shell; C₁₄-C₂₂ midchain branched alcohols; C₁₄-C₂₂ midchain branched alkyl ethoxylates, BAE_x, wherein *x* is from 1 to 30. The nonionic ethoxylated alcohol surfactant herein may further comprise residual alkoxylation catalyst, which may be considered residue from the reaction or an impurity. It may further comprise various impurities or by-products of the alkoxylation reaction. The impurities may vary depending on the catalyst used and the conditions of the reaction. Impurities include alkyl ethers, e.g., dialkyl ethers, such as, didodecyl ether, glycols, e.g., diethylene glycol, triethylene glycol, pentaethylene glycol, other polyethylene glycols.

The nonionic ethoxylated alcohol may be a narrow range ethoxylated alcohol. A narrow range ethoxylated alcohol may have the following general formula (I):

where R is selected from a saturated or unsaturated, linear or branched, C_8 - C_{20} alkyl group and where greater than 90% of n is $0 \le n \le 15$. In addition, the average value of n can be between about 6 to about 10, where less than about 10% by weight of the alcohol ethoxylate are ethoxylates having n< 7 and between 10% and about 20% by weight of the alcohol ethoxylate are ethoxylates having n=8.

The composition may comprise an average value of n of about 10. The composition may have the following ranges for each of the following n: n = 0 of up to 5%, each of n = 1, 2, 3, 4, 5 of up to 2%, n = 6 of up to 4%, n = 7 of up to 10%, n = 8 of between 12% and 20%, n = 9 of between 15% and 25%, n = 10 of between 15% to 30%, n = 11 of between 10% and 20%, n = 12 of up to 10%, and n > 12 at up to 10%. The composition may have n = 9 to 10 of between 30% and 70%. The composition may have greater than 50% of its composition made up of n = 8 to 11.

30

5

10

15

20

The alcohol ethoxylates described herein are typically not single compounds as suggested by their general formula (I), but rather, they comprise a mixture of several homologs having varied polyalkylene oxide chain length and molecular weight. Among the homologs, those with the number of total alkylene oxide units per mole of alcohol closer to the most prevalent alkylene oxide adduct are desirable; homologs whose number of total alkylene oxide units is much lower or much higher than the most prevalent alkylene oxide adduct are less desirable. In other words, a "narrow range" or "peaked" alkoxylated alcohol composition is desirable. A "narrow range" or "peaked" alkoxylated alcohol composition refers to an alkoxylated alcohol composition having a narrow distribution of alkylene oxide addition moles.

A "narrow range" or "peaked" alkoxylated alcohol composition may be desirable for a selected application. Homologs in the selected target distribution range may have the proper lipophilic-hydrophilic balance for a selected application. For example, in the case of an ethoxylated alcohol product comprising an average ratio of 5 ethylene oxide (EO) units per molecule, homologs having a desired lipophilic-hydrophilic balance may range from 2EO to 9EO. Homologs with shorter EO chain length (<2EO) or longer EO chain length (>9EO) may not be desirable for the applications for which a =5 EO/alcohol ratio surfactant is ordinarily selected since such longer and shorter homologs are either too lipophilic or too hydrophilic for the applications utilizing this product. Therefore, it is advantageous to develop an alkoxylated alcohol having a peaked distribution.

The narrow range alkoxylated alcohol compositions of the disclosure may have an average degree of ethoxylation ranging from about 0 to about 15, such as, for example, ranging from about 4 to about 14, from about 5-10, from about 8-11, and from about 6-9. The narrow range alkoxylated alcohol compositions of the disclosure may have an average degree of ethoxylation of 10. The narrow range alkoxylated alcohol compositions of the disclosure may have an average degree of ethoxylation of 9. The narrow range alkoxylated alcohol compositions of the disclosure may have an average degree of ethoxylation of 5.

Non-limiting examples of cationic surfactants include: the quaternary ammonium surfactants, which can have up to 26 carbon atoms include: alkoxylate quaternary ammonium (AQA) surfactants; dimethyl hydroxyethyl quaternary ammonium surfactants; dimethyl hydroxyethyl lauryl ammonium chloride; polyamine cationic surfactants; cationic ester surfactants; and amino surfactants, such as amido propyldimethyl amine (APA). The compositions of the present disclosure may be substantially free of cationic surfactants and/or of

5

10

15

20

25

WO 2023/102341 PCT/US2022/080398 25

surfactants that become cationic below a pH of 7 or below a pH of 6, as cationic surfactants may negatively interact with other components, such as anionic surfactants.

Examples of zwitterionic surfactants include derivatives of secondary and tertiary amines, derivatives of heterocyclic secondary and tertiary amines, or derivatives of quaternary ammonium, quaternary phosphonium or tertiary sulfonium compounds. The zwitterionic surfactants may comprise betaines, including alkyl dimethyl betaine, cocodimethyl amidopropyl betaine, and C₈ to C₁₈ (for example from C₁₂ to C₁₈) amine oxide and sulfo and hydroxy betaines, such as N-alkyl-N,N-dimethylammino-1-propane sulfonate where the alkyl group can be from C₈ to C₁₈ or from C₁₀ to C₁₄.

10 Detergent Adjunct

5

15

20

25

30

The liquid detergent composition can comprise one or more adjunct ingredients at a level, for example, of about 0.1% to about 50%. Adjunct ingredients can include, for example, color care agents; organic solvents; aesthetic dyes; hueing dyes; leuco dyes; opacifiers such as those commercially available under the Acusol tradename, brighteners including FWA49, FWA15, and FWA36; dye transfer inhibitors including PVNO, PVP and PVPVI dye transfer inhibitors; builders including citric acid- and fatty acids; chelants; enzymes; perfume capsules; preservatives; antioxidants including sulfite salts such as potassium sulphite or potassium bisulphite salts and those commercially available under the Ralox brand name, antibacterial and anti-viral agents including 4.4'-dichloro 2-hydroxydiphenyl ether such as Tinosan HP100 available from the BASF company; anti-mite actives such as benzyl benzoate; structuring agents including hydrogenated castor oil; silicone based anti-foam materials; electrolytes including inorganic electrolytes such as sodium chloride, potassium chloride, magnesium chloride, and calcium chloride, and related sodium, potassium, magnesium and calcium sulphate salts, as well as organic electrolytes such as sodium, potassium, magnesium and calcium salts of carbonate, bicarbonate, carboxylates such as formate, citrate and acetate; pH trimming agents including sodium hydroxide, hydrogen chloride, and alkanolamines including monoethanolamine, diethanolamine, triethanolamine, and monoisopropanolamine; a probiotic; a hygiene agent such as zinc ricinoleate, thymol, quaternary ammonium salts such as Bardac®, polyethylenimines (such as Lupasol® from BASF) and zinc complexes thereof, silver and silver compounds, a cationic biocide including octyl decyl dimethyl ammonium chloride, dioctyl dimethyl ammonium chloride, didecyl dimethyl ammonium chloride, dispersant, cleaning polymer, glucan, or a mixture thereof. For example, the detergent adjunct comprises an enzyme, an enzyme stabilizer, a builder, a hueing agent, anti-soil redeposition agent, a bleach, or a combination thereof.

26

The organic solvent can include an alcohol and/or a polyol. For example, the organic solvent can comprise ethanol, propanol, isopropanol, a sugar alcohol, a glycol, a glycol ether, or a combination thereof. The organic solvent can comprise polyethylene glycol, especially low molecular weight polyethylene glycols such as PEG 200 and PEG 400; diethylene glycol; glycerol; 1,2-propanediol; polypropylene glycol including dipropylene glycol and tripropylene glycol and low molecular weight polypropylene glycols such as PPG400; or a mixture thereof.

The chelant can comprise, for example, EDDS, HEDP, GLDA, DTPA, DTPMP, DETA, EDTA, MGDA or a mixture thereof. The chelant can be biodegradable. Biodegradable chelants can include, for example, NTA, IDS, EDDG, EDDM, HIDS, HEIDA, HEDTA, DETA, or a combination thereof.

The enzyme can comprise, for example, protease, amylase, cellulase, mannanase, lipase, xyloglucanase, pectate lyase, nuclease enzyme, or a mixture thereof.

Cleaning polymers can include, for example, those which can help clean stains or soils on clothing and/or help prevent those soils from redepositing on clothing during the wash. Examples are optionally modified carboxymethylcellulose, modified polyglucans, poly(vinyl-pyrrolidone), poly (ethylene glycol), poly(vinyl alcohol), poly(vinylpyridine-N-oxide), poly(vinylimidazole), polycarboxylates such as polyacrylates, maleic/acrylic acid copolymers and lauryl methacrylate/acrylic acid co-polymers.

The composition may comprise one or more amphiphilic cleaning polymers. Such polymers have balanced hydrophilic and hydrophobic properties such that they remove grease particles from fabrics and surfaces. Suitable amphiphilic alkoxylated grease cleaning polymers comprise a core structure and a plurality of alkoxylate groups attached to that core structure. These may comprise alkoxylated polyalkylenimines, especially ethoxylated polyethylene imines or polyethyleneimines having an inner polyethylene oxide block and an outer polypropylene oxide block. Typically, these may be incorporated into the compositions of the invention in amounts of from 0.005 to 10 wt%, generally from 0.5 to 8 wt%.

Water

The detergent composition may also include water. Water can be present, for example, at a level of about 5% to about 95%, by weight of the composition.

30 pH

5

10

15

20

25

The detergent composition may have a pH of about 5.0 to about 12, preferably 6.0-10.0, more preferably from 8.0 to 10. wherein the pH of the detergent composition is measured as a 10% dilution in demineralized water at 20°C.

Viscosity

5

10

20

25

A liquid detergent composition can be in the form of an aqueous solution or uniform dispersion or suspension. Such a solution, dispersion or suspension will be acceptably phase stable. A liquid detergent composition can have a viscosity from 1 to 1500 centipoises (1-1500 mPa*s), more preferably from 100 to 1000 centipoises (100-1000 mPa*s), and most preferably from 200 to 500 centipoises (200-500 mPa*s) at 20s-1 and 21°C. Viscosity can be determined by conventional methods. Viscosity may be measured using an AR 550 rheometer from TA instruments using a plate steel spindle at 40 mm diameter and a gap size of 500 μm. The high shear viscosity at 20s-1 and low shear viscosity at 0.05-1 can be obtained from a logarithmic shear rate sweep from 0.1-1 to 25-1 in 3 minutes time at 21°C. The preferred rheology described therein may be achieved using internal existing structuring with detergent ingredients or by employing an external rheology modifier. More preferably the laundry care compositions, such as detergent liquid compositions have a high shear rate viscosity of from about 100 centipoise to 1500 centipoise, more preferably from 100 to 1000 cps.

15 Composition Making

The liquid compositions can be prepared, for example, by combining the components thereof in any convenient order and by mixing, e.g., agitating, the resulting component combination to form a phase stable liquid laundry care composition. In a process for preparing such compositions, a liquid matrix can be formed containing at least a major proportion, or even substantially all, of the liquid components, e.g., nonionic surfactant, the non-surface-active liquid carriers and other optional liquid components, with the liquid components being thoroughly admixed by imparting shear agitation to this liquid combination. For example, rapid stirring with a mechanical stirrer may usefully be employed. While shear agitation is maintained, substantially all of any anionic surfactants and the solid form ingredients can be added. Agitation of the mixture is continued, and if necessary, can be increased at this point to form a solution or a uniform dispersion of insoluble solid phase particulates within the liquid phase. After some or all of the solid-form materials have been added to this agitated mixture, particles of any enzyme material to be included, e.g., enzyme prills, are incorporated. As a variation of the composition preparation procedure hereinbefore described, one or more of the solid components may be added to the agitated mixture as a solution or slurry of particles premixed with a minor portion of one or more of the liquid components. After addition of all of the composition components, agitation of the mixture is continued for a period of time sufficient to form compositions having

the requisite viscosity and phase stability characteristics. Frequently this will involve agitation for a period of from about 30 to 60 minutes.

COMBINATIONS

- 1. A liquid detergent composition comprising:
- a) from about 1% to about 30%, by weight of the composition of a first surfactant consisting essentially of a mixture of surfactant isomers of Formula 1 and surfactants of Formula 2:

$$CH_2$$
— X

I

10 Formula 1: CH_3 — $(CH_2)_m$ — CH — $(CH_2)_n$ — CH_3 6 ≤ m ≤ 11; 0 ≤ n ≤ 5:

Formula 2:
$$CH_3$$
— $(CH_2)_{m+n+3}$ — X

wherein from about 50% to about 100% by weight of the first surfactant are isomers having m+n=11; wherein between about 25% to about 50% of the mixture of surfactant isomers of Formula 1 have n=0; wherein from about 0.001% to about 25% by weight of the first surfactant are surfactants of Formula 2; and wherein X is a hydrophilic moiety;

- b) from about 0.1% to about 5% by weight of the composition of an encapsulate comprising a shell and a core, wherein the shell comprises a polyacrylate and the core comprises a perfume; and
 - c) a detergent adjunct.
- 2. The liquid detergent composition of 1, wherein the liquid detergent composition has a greater dry fabric odor, preferably 10% greater dry fabric odor, versus the combination of scores of a first reference composition comprising the first surfactant but not the encapsulate and a second reference composition comprising the encapsulate but not the first surfactant.
- 3. The liquid detergent composition of any of 1 2, wherein the liquid detergent composition has a greater crushed fabric odor, preferably 10% greater crushed fabric odor, versus the combination of scores of a first reference composition comprising the

30

15

20

- first surfactant but not the encapsulate and a second reference composition comprising the encapsulate but not the first surfactant.
- 4. The liquid detergent composition of any of 1-3, wherein the ratio by weight of the first surfactant to the encapsulate is from about 300:1 to about 2:1; preferably from about 35:1 to about 5:1.
- 5. The liquid detergent composition of any of 1-4, further comprising an additional surfactant.
- 6. The liquid detergent composition of 6, wherein the additional surfactant comprises linear alkyl benzene sulfonate, alkyl sulfate, alkyl ethoxylated sulfate, a nonionic surfactant, or a combination thereof.
- 7. The liquid detergent composition of any of 1-6, wherein the detergent adjunct comprises an enzyme, an enzyme stabilizer, a builder, a hueing agent, anti-soil redeposition agent, a bleach, or a combination thereof.
- 8. The liquid detergent composition of any of 1-7, wherein between about 15% to about 40% by weight of the first surfactant, of the mixture of surfactant isomers of Formula 1 have n = 1.
 - The liquid detergent composition of any of 1-8, wherein about 60% to about 90% by weight of the first surfactant, of the mixture of surfactant isomers of Formula 1 have n <
 3.
- 20 10. The liquid detergent composition of any of 1-9, wherein about 90% to about 100%, by weight of the first surfactant, of surfactant isomers have an m+n =11.
 - 11. The liquid detergent composition of any of 1-10, wherein X comprises a sulfate.
 - 12. The liquid detergent composition of any of 1-11, further comprising from about 10% to about 90% water, preferably from about 15% to about 85%.
- The liquid detergent composition of any of 1-12, wherein the encapsulate shell is about 60 nanometers to about 200 nanometers thick.
 - 14. The liquid detergent composition of any of 1-13, wherein the shell wall comprises about 70% or more of an aliphatic urethane acrylate polymer, preferably about 90% or more.

5

EXAMPLES

5

10

15

20

EXAMPLE 1: Preparation of a Branched C15 Alcohol Product

The homogeneous rhodium organophosphorus catalyst used in this example is prepared in a high stainless steel stirred autoclave. To the autoclave was added 0.027 pressure. wt.% Rh(CO)2ACAC ((Acetylacetonato)dicarbonylrhodium(I)), 1.36wt.% tris butylphenyl) phosphite ligand and 98.62 wt.% Synfluid® PAO 4 cSt (Chevron Phillips Chemical Company LP, P.O. Box 4910, The Woodlands, TX 77387-4910, phone (800) 231-3260) inert solvent. The mixture was heated at 80°C in the presence of a CO/H2 atmosphere and 2 bar(g) pressure for four hours to produce the active rhodium catalyst solution (109 ppm rhodium, P:Rh molar ratio = 20). A C14 linear alpha olefin feedstock (1-Tetradecene) from the Chevron Phillips Chemical Company LP, (AlphaPlus® 1-Tetradecene by Chevron Phillips Chemical Company LP, P.O. Box 4910, The Woodlands, TX 77387-4910, phone (800) 231-3260) was added. The resulting mixture had a rhodium concentration of approximately 30 ppm. The 1-tetradecene linear alpha olefin was then isomerized at 80°C in the presence of a CO/H2 atmosphere and 1 bar(g) pressure for 12 hours. The isomerized olefin then hydroformylated at 70°C in the presence of a CO/H2 atmosphere and 20 bar(g) pressure for 8 hours. The resulting reaction product was flash distilled at 150-160°C and 25 millibar to recover the rhodium catalyst solution as a bottoms product and recover a branched C15 Aldehyde overheads product. The recovered rhodium catalyst solution was then used again to complete a second 1-tetradecene batch isomerization (4 hours) and hydroformylation (6 hours). The resulting C15 aldehyde products from the two batches were combined to give a branched C15 Aldehyde product comprising:

| | Weight % | |
|----|-----------------------|----------|
| | 1-Pentadecanal | 12.1% |
| 25 | 2-Methyl-tetradecanal | 34.1% |
| | 2-Ethyl-tridecanal | 21.9% |
| | 2-Propyl-dodecanal | 14.0% |
| | 2-Butyl-undecanal | 8.6% |
| | 2-Pentyl-decanal | 9.0% |
| 30 | +2-hexyl-decana | <u>1</u> |
| | TOTAL | 99.6% |

The weight % branching in the branched C15 aldehyde product was 87.8%.

31

The branched C15 aldehyde product was hydrogenated in a high pressure, Inconel 625 stirred autoclave at 150C and 20 bar(g) hydrogen pressure. The hydrogenation catalyst used was a Raney® Nickel 3111 (W. R. Grace & Co., 7500 Grace Drive, Columbia, MD 21044, US, phone 1-410-531-4000) catalyst used at a 0.25wt.% loading. The aldehyde was hydrogenated for 10 hours and the resultant reaction mixture was filtered to produce a branched C15 alcohol product comprising:

| | Weight % | |
|----|-----------------------|-------|
| | 1-Pentadecanol | 13.7% |
| | 2-Methyl-tetradecanol | 32.6% |
| 10 | 2-Ethyl-tridecanol | 21.7% |
| | 2-Propyl-dodecanol | 12.4% |
| | 2-Butyl-undecanol | 8.0% |
| | 2-Pentyl-decanol + | 9.0% |
| | 2-hexyl-decanol | |
| 15 | Other | 2.7% |

The weight % 2-alkyl branching in the branched C15 alcohols product was 83.6%.

Example 2. Synthesis of Narrow Branched Pentadecanol (C15) Sulfate using a Falling Film Sulfation Reactor (branched alkyl sulfate Example Z)

The alcohol from Example 1 is sulfated in a falling film using a Chemithon single 15 mmx2 m tube reactor using SO3 generated from a sulfur burning gas plant operating at 5.5 lb/hr sulfur to produce 3.76% SO3 on a volume basis. Alcohol feed rate is 17.4 kg/hour and feed temperature was 83 F. Conversion of the alcohol to alcohol sulfate acid mix was achieved with 97% completeness. Neutralization with 50% sodium hydroxide is completed at ambient process temperature to 0.54% excess sodium hydroxide. 30 gallons of sodium neutralized C15 narrow branched Alcohol Sulfate paste. Analyses by standard Cationic SO3 titration method determines final average product activity to be 74.5%. The average unsulfated level is 2.65% w/w.

25

20

Table 5: Alkyl chain distribution of C15 Alkyl Sulfates based on starting distribution of alcohol

| | | C15 Alcohol | Branched |
|-----------------------------|------------|-------------|-------------------------|
| | | from | alkyl sulfate Example Z |
| | Neodol® 5 | US9493725 | made from |
| | (ex Shell) | (ex Sasol) | Example 1 C15 Alcohol |
| Linear C15 * | 79.3 | 8.6 | 13.7 |
| 2-Alkyl Branched C15 | 17.5 | 89.5 | 83.6 |
| Other* | 3.2 | 1.9 | 2.7 |
| 2-methyl* | 7.0 | 19.0 | 32.6 |
| 2-ethyl* | 2.8 | 12.0 | 21.7 |
| 2-propyl* | 1.9 | 12.7 | 12.4 |
| 2-buty1* | 2.0 | 14.6 | 8.0 |
| 2-pentyl+2-hexyl* | 3.8 | 31.2 | 9.0 |
| 2-Alkyl Branch Distribution | | | |
| 2-methy1** | 39.9% | 21.2% | 38.9% |
| 2-ethyl** | 16.2% | 13.4% | 25.9% |
| 2-propyl** | 10.7% | 14.2% | 14.9% |
| 2-buty1** | 11.3% | 16.3% | 9.5% |
| 2-pentyl + 2-hexyl** | 21.9% | 34.9% | 10.7% |

^{*} by weight of starting alcohol

** by weight of 2-alkyl branched C15 alcohol

Formulation Examples

| | | Comparative Comp. A (chassis) | Comparative Comp. B | Comparative Comp. C | Comparative Comp. D | Comparative Comp. E |
|-------------------------------|-------|-------------------------------------|-------------------------|------------------------|------------------------|------------------------|
| Raw Material | | | % active in formulation | | | |
| PAC PMC ¹ | | - | 0.13 | 0.26 | 0.51 | - |
| Branched sulfate ² | alkyl | - | - | - | - | 1.98 |

| AES ³ | 2.25 | 2.25 | 2.25 | 2.25 | 2.25 |
|---|---------|---------|---------|---------|---------|
| HLAS ⁴ | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 |
| SLS ⁵ | 0.88 | 0.88 | 0.88 | 0.88 | 0.88 |
| NI^6 | 5.14 | 5.14 | 5.14 | 5.14 | 5.14 |
| Chelant ⁷ | 0.24 | 0.24 | 0.24 | 0.24 | 0.24 |
| Citric acid ⁸ | 0.61 | 0.61 | 0.61 | 0.61 | 0.61 |
| Protease ⁹ | 0.06 | 0.06 | 0.06 | 0.06 | 0.06 |
| Borate ¹⁰ | 1.45 | 1.45 | 1.45 | 1.45 | 1.45 |
| Ethoxylated polyethyleneimine ¹¹ | 3.28 | 3.28 | 3.28 | 3.28 | 3.28 |
| Suds suppressor ¹² | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 |
| Hydrogenated Castor oil | 0.14 | 0.14 | 0.14 | 0.14 | 0.14 |
| Misc (water, etc.) | Balance | Balance | Balance | Balance | Balance |

| | Comparative Comp. F | Comparative Comp. G | Inventive Comp. 1 | Inventive Comp. 2 | Inventive Comp. 3 |
|---|------------------------|------------------------|----------------------|----------------------|----------------------|
| Raw Material | | | % active in formule | ation | |
| PAC PMC ¹ | - | - | 0.13 | 0.26 | 0.26 |
| Branched alkyl sulfate ² | 3.97 | 7.93 | 1.98 | 1.98 | 3.97 |
| AES ³ | 2.25 | 2.25 | 2.25 | 2.25 | 2.25 |
| HLAS ⁴ | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 |
| SLS ⁵ | 0.88 | 0.88 | 0.88 | 0.88 | 0.88 |
| NI ⁶ | 5.14 | 5.14 | 5.14 | 5.14 | 5.14 |
| Chelant ⁷ | 0.24 | 0.24 | 0.24 | 0.24 | 0.24 |
| Citric acid ⁸ | 0.61 | 0.61 | 0.61 | 0.61 | 0.61 |
| Protease ⁹ | 0.06 | 0.06 | 0.06 | 0.06 | 0.06 |
| Borate ¹⁰ | 1.45 | 1.45 | 1.45 | 1.45 | 1.45 |
| Ethoxylated polyethyleneimine ¹¹ | 3.28 | 3.28 | 3.28 | 3.28 | 3.28 |
| Suds suppressor ¹² | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 |
| Hydrogenated Castor oil | 0.14 | 0.14 | 0.14 | 0.14 | 0.14 |
| Misc (water, etc.) | Balance | Balance | Balance | Balance | Balance |

| | Inventive Comp. 4 | Inventive Comp. 5 | | |
|-------------------------------------|-------------------------|----------------------|--|--|
| Raw Material | % active in formulation | | | |
| PAC PMC ¹ | 0.26 | 0.51 | | |
| Branched alkyl sulfate ² | 7.93 | 7.93 | | |
| AES^3 | 2.25 | 2.25 | | |
| HLAS ⁴ | 10.00 | 10.00 | | |
| SLS ⁵ | 0.88 | 0.88 | | |
| NI ⁶ | 5.14 | 5.14 | | |
| Chelant ⁷ | 0.24 | 0.24 | | |
| Citric acid ⁸ | 0.61 | 0.61 | | |
| Protease ⁹ | 0.06 | 0.06 | | |

| Borate ¹⁰ | 1.45 | 1.45 |
|---|---------|---------|
| Ethoxylated polyethyleneimine ¹¹ | 3.28 | 3.28 |
| Suds suppressor ¹² | 0.20 | 0.20 |
| Hydrogenated Castor oil | 0.14 | 0.14 |
| Misc (water, etc.) | Balance | Balance |

¹ PAC PMC CN975 available from Sartomer Inc.; ² branched alkyl sulfate Example Z; ³ C12-15EO2.5S AlkylethoxySulfate where the alkyl portion of AES has a weight average molecular weight of 211 to 218 daltons, available from P&G Chemicals; ⁴ High C12 about 96% Linear Alkyl Benzene Sulfonate sourced from P&G Chemicals; ⁵ C12/C14 Alkyl Sulfate sourced from P&G Chemicals; ⁶ Surfonic L24-9 commercially available from Huntsman; ⁷ DISSOLVINE GL-47-S commercially available from AkzoNobel; ⁸ Citrosol 502 commercially available from Archer Daniels Midland; ⁹ Preferenz commercially available from DuPont; ¹⁰ Disodium tetraborate pentahydrate commercially sourced from Univar Solutions; ¹¹ PE-20 commercially available from BASF; ¹² Suds suppressor DOWSIL AF-8017 commercially available from Dow

The comparative and inventive examples are prepared by combining all raw materials to achieve Comparative Composition A, with exception of not adding suds suppressor, hydrogenated castor oil, and all of the water to leave space (referred to as a hole) to add in the branched alkyl sulfate and encapsulates for Comparative Compositions B-G and Inventive Compositions 1-5. The following raw materials were mixed rapidly to achieve a vortex with a mixing impeller for about 60 minutes: water, solvent, surfactant, stabilizer, borax, neutralizer, builder, chelant, polymer, and enzyme to result in a stable one phase liquid.

To make Comparative Compositions B-G and Inventive Compositions 1-5, the combinations of branched alkyl sulfate, encapsulates suds suppressor, hydrogenated castor oil, and water to balance were added on top of Comparative Composition A (with the hole) to achieve the desired levels. Consistent pH of 8.4-8.7 was measured between all tested formulas. Methods

Fabric Headspace Analysis Method

The method involves the use of a tergotometer to simulate the washing of fabrics in a washing machine. Terry fabrics were ordered from Calderon (Indianapolis, IN, USA). Fabrics were stripped using 2 wash and rinse cycles using 48 g AATCC detergent in 140°F and soft water and 2 wash and rinse cycles without product in 140°F soft water. Preconditioned fabrics were generated using 3 detergent/LFE cycles using 85 g nil-perfume/perfume encapsulate

5

10

15

20

detergent and 48.5 g liquid nil-perfume/perfume encapsulate fabric enhancer. Each cycle is run with 7 gpg water in 90°F wash/60°F rinse.

Wash tests consisted of four internal and 3 external replicates for each treatments A-L described below (Table 2). Each wash test contained a ratio of 18.93L water to 48.48g liquid detergent test compositions, test fabrics at 25°C and 7 US gpg, agitated for 12 minutes, rinsed in 25°C water at 7 US gpg for 3 minutes, and spun dry. After the rinse, fabrics were dried at 145°F for 30 minutes. Before the fabrics were analysed, the fabrics were exposed to 73°F and 50% humidity for 4 hours. GCMS headspace analysis was used on each fabric sample, with the method described below.

Fabric headspace analysis is performed using Solid-phase Micro Extraction Gas Chromatography Mass Spectrometry (SPME GC-MS) described below. Typically, greater perfume intensity (as measured by headspace analysis) correlates with higher concentrations of perfume encapsulates on fabric. Perfume encapsulate headspace analysis is carried out on treated 100% cotton terry towels (30.5cm x 30.5cm, RN37000ITL, Calderon Textiles, LLC, Indianapolis, IN, USA) that have been prepared and treated according to the fabric preparation method that is described above.

Headspace analysis is done on four treated fabrics from three different wash cycles for a total of twelve fabrics. Approximately 8 1 cm dots of test fabrics are placed into a 20mL headspace sample vial (#24694, available from Restek, Bellefonte, PA), and the vial is capped (#093640-094-00 available from Gerstel, Linthicum, MD). Dry fabrics are equilibrated for 4 hours in a constant temperature and humidity room at 21C and 50% RH prior to capping. Crushed fabrics were uncapped at the apparatus, followed by lowering a rod with weight equal to 67psi. Pressure was applied for 10 seconds, and then waited 10 seconds before capping the vial.

The samples vials are then loaded onto a Gerstel MPS2 Autosampler (Gerstel Inc., Linthicum, MD, USA). Prior to the headspace analysis, each sample is pre-conditioned in the machine at 65°C for 10 minutes. Headspace is extracted onto an Agilent 7890B/5977A GC-MS system (Agilent Technologies, Santa Clara, CA, USA) equipped with a Supelco 50/30 micrometer DVB/CAR/PDMS 23Ga. Solid Phase Micro Extraction fiber (Supelco Inc., Bellefonte, PA, USA). GC analysis is conducted on a non-polar capillary column (DB-5MS UI, 30 meters nominal diameter, 0.25 millimeter nominal diameter, 25 micrometer thickness) and the headspace constituents (i.e. the perfume raw materials) are monitored by Mass Spectrometry (EI, 70eV detector). Headspace intensity is calculated utilizing a single point calibration of the perfume raw materials. The total headspace concentration for each vial is calculated from the

5

10

15

20

25

sum of the concentration of each detected perfume raw material, and the headspace is averaged for the twelve treated fabrics. Headspace improvement may be determined relative to the reference treatment.

The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as "40 mm" is intended to mean "about 40 mm."

Every document cited herein, including any cross referenced or related patent or application and any patent application or patent to which this application claims priority or benefit thereof, is hereby incorporated herein by reference in its entirety unless expressly excluded or otherwise limited. The citation of any document is not an admission that it is prior art with respect to any invention disclosed or claimed herein or that it alone, or in any combination with any other reference or references, teaches, suggests or discloses any such invention. Further, to the extent that any meaning or definition of a term in this document conflicts with any meaning or definition of the same term in a document incorporated by reference, the meaning or definition assigned to that term in this document shall govern.

While particular embodiments of the present invention have been illustrated and described, it would be obvious to those skilled in the art that various other changes and modifications can be made without departing from the spirit and scope of the invention. It is therefore intended to cover in the appended claims all such changes and modifications that are within the scope of this invention.

5

10

15

WO 2023/102341 37

PCT/US2022/080398

CLAIMS

What is claimed is:

- 1. A liquid detergent composition comprising:
 - a) from 1% to 30%, by weight of the composition of a first surfactant consisting essentially of a mixture of surfactant isomers of Formula 1 and surfactants of Formula 2:

$$CH_2 - X$$

$$I$$
 Formula 1: $CH_3 - (CH_2)_m - CH - (CH_2)_n - CH_3$ $6 \le m \le 11$; $0 \le n \le 5$;

Formula 2:
$$CH_3$$
— $(CH_2)_{m+n+3}$ — X

wherein from 50% to 100% by weight of the first surfactant are isomers having m+n=11; wherein between 25% to 50% of the mixture of surfactant isomers of Formula 1 have n=0; wherein from 0.001% to 25% by weight of the first surfactant are surfactants of Formula 2; and wherein X is a hydrophilic moiety;

- b) from 0.1% to 5% by weight of the composition of an encapsulate comprising a shell and a core, wherein the shell comprises a polyacrylate and the core comprises a perfume; and
 - c) a detergent adjunct.
- 2. The liquid detergent composition of claim 1, wherein the liquid detergent composition has a greater dry fabric odor, preferably 10% greater dry fabric odor, versus the combination of scores of a first reference composition comprising the first surfactant but not the encapsulate and a second reference composition comprising the encapsulate but not the first surfactant.
- 3. The liquid detergent composition of any of claims 1 2, wherein the liquid detergent composition has a greater crushed fabric odor, preferably 10% greater crushed fabric odor, versus the combination of scores of a first reference composition comprising the first surfactant but not the encapsulate and a second reference composition comprising the encapsulate but not the first surfactant.

- 4. The liquid detergent composition of any of claims 1-3, wherein the ratio by weight of the first surfactant to the encapsulate is from 300:1 to 2:1; preferably from 35:1 to 5:1.
- 5. The liquid detergent composition of any of claims 1-4, further comprising an additional surfactant.
- 6. The liquid detergent composition of claim 5, wherein the additional surfactant comprises linear alkyl benzene sulfonate, alkyl sulfate, alkyl ethoxylated sulfate, a nonionic surfactant, or a combination thereof.
- 7. The liquid detergent composition of any of claims 1-6, wherein the detergent adjunct comprises an enzyme, an enzyme stabilizer, a builder, a hueing agent, anti-soil redeposition agent, a bleach, or a combination thereof.
- 8. The liquid detergent composition of any of claims 1-7, wherein between 15% to 40% by weight of the first surfactant, of the mixture of surfactant isomers of Formula 1 have n = 1.
- 9. The liquid detergent composition of any of claims 1-8, wherein 60% to 90% by weight of the first surfactant, of the mixture of surfactant isomers of Formula 1 have n < 3.
- 10. The liquid detergent composition of any of claims 1-9, wherein 90% to 100%, by weight of the first surfactant, of surfactant isomers have an m+n=11.
- 11. The liquid detergent composition of any of claims 1-10, wherein X comprises a sulfate.
- 12. The liquid detergent composition of any of claims 1-11, further comprising from 10% to 90% water, preferably from 15% to 85%.
- 13. The liquid detergent composition of any of claims 1-12, wherein the encapsulate shell is 60 nanometers to 200 nanometers thick.
- 14. The liquid detergent composition of any of claims 1-13, wherein the shell wall comprises 70% or more of an aliphatic urethane acrylate polymer, preferably 90% or more.