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㉓ Concentrated, liquid, pourable composition.

㉔ A concentrated, liquid, pourable composition comprising:

- a) from 50 to 95% by weight of an alkali or earth alkali metal or ammonium salt or a salt of an organic base of an internal olefin sulphonic acid, having from 8 to 26 carbon atoms, these internal olefin sulphonates having at least 25% by weight of beta-hydroxy sulphonates;
- b) from 4 to 49% by weight of water; and
- c) from 1 to 46% by weight of a lower alcohol, a nonionic surfactant, a polyethylene glycol, a polypropylene glycol, a salt-tolerant anionic surfactant, or a mixture thereof.

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The invention relates to a concentrated, liquid, pourable composition comprising an alkali or earth alkali metal or ammonium salt or a salt of an organic base of an internal olefin sulphonic acid, having 8 to 26 carbon atoms.

In the European patent application No. 89203337 has been described a detergent composition comprising an alkali or earth alkali metal or ammonium salt or a salt of an organic base of an internal olefin sulphonic acid, having from 8 to 26 carbon atoms, and containing at least 25% by weight of beta-hydroxysulphonate, calculated on the total amount of sulphonate. Especially compositions with 50 to 90% by weight of beta-hydroxysulphonate are used.

There is further described that the compositions may contain other surfactants of anionic, nonionic, amphoteric or cationic type and depending upon the formulations to be used the compositions may contain builders, sequestring agents, bleaching agents, bleach activators, greyness-preventing agents, soil release polymers, foam control agents, fluorescent whiteners, enzymes and perfumes. The compositions are thus suitable for a number of applications such as solid and liquid laundry detergents, dishwash detergents, cleaning agents, liquid soaps, shampoos and liquid scouring agents.

There have now been found concentrated, liquid and pourable compositions which have advantages over the compositions, disclosed in European patent application No. 89203337.

The invention relates to a concentrated liquid, pourable composition comprising:

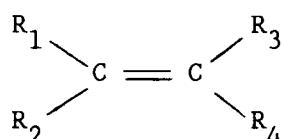
- a) from 50 to 95% by weight of an alkali or earth alkali metal or ammonium salt or a salt of an organic base of an internal olefin sulphonic acid, having from 8 to 26 carbon atoms, these internal olefin sulphonates having at least 25% by weight of beta-hydroxy sulphonates;
- b) from 4 to 49% by weight of water; and
- c) from 1 to 46% by weight of a lower alcohol, a nonionic surfactant, a polyethylene glycol, a polypropylene glycol, a salt-tolerant anionic surfactant, or a mixture thereof.

The inventive compositions are easily storageable, handable and transportable. Furthermore, they may be easily spray-dried or converted into less concentrated compositions.

Component a), the internal olefin sulphonate, contains at least 25% by weight of beta-hydroxy sulphonate, preferably at least 50%, more preferably more than 70% by weight of beta-hydroxy sulphonate. Preferred internal olefin sulphonic acids contain from 13 to 24 carbon atoms. The sulphonic acids may be present in the form of their salts with sodium, potassium, ammonium or an organic base.

The internal olefin sulphonates, containing beta-hydroxy sulphonates, are generally prepared by reacting in a film reactor an internal olefin having from 8 to 26 carbon atoms with a sulphonating agent, in a mol ratio of sulphonating agent to internal olefin of 1.3 to 0.9, preferably 1.15 to 0.95, while cooling the reactor with a cooling means having a temperature not exceeding 35 °C and allowing the reaction product of the sulphonating step to neutralize and hydrolyze.

The internal olefins used as starting material are those having 8 to 26 carbon atoms, preferably 13 to 24 carbon atoms. Examples of the internal olefins are those having the general formula:



wherein each of R₁, R₂, R₃ and R₄ independently are linear or branched alkyl groups or hydrogen and the total carbon number of R₁, R₂, R₃ and R₄ is from 6 to 24, and wherein at least one of R₁ and R₂ and one of R₃ and R₄ is an alkyl group.

In the process for preparing internal olefin sulphonates the neutralization/hydrolysis step is carried out directly after the sulphonating step. An ageing step is consequently avoided or minimized.

The sulphonation may be carried out batchwise, semi-continuously or continuously, preferably continuously.

The neutralization/hydrolysis is preferably carried out continuously and at a temperature of 20 to 50 °C, preferably 30 to 45 °C. Hydrolysis is preferably carried out continuously and at temperatures of 140-190 °C.

For the neutralization/hydrolysis an aqueous base is generally used. To prepare concentrated products, the amount of water is regulated accordingly.

Component c) may be included in the composition at the same time when water is added. This third component is a lower alcohol, a nonionic surfactant, a polyethylene glycol, a polypropylene glycol, a salt-tolerant anionic surfactant or a mixture thereof.

Examples of lower alcohols are methanol, ethanol, propanol, iso-propanol, butanol, iso-butanol, tert.butanol, a pentanol or a hexanol, ethylene glycol, propylene glycol, a linear or branched C₁ to C₆ mono-alkylether of mono- or di-ethylene glycol, a linear or branched C₁ to C₆ mono-alkylether of mono- or di-propylene glycol, or a mixture thereof.

5 Examples of nonionic surfactants are C₇ to C₁₈-alcohols that are alkoxylated with ethylene oxide and/or propylene oxide with a minimum degree of alkoxylation of 2.

Further examples of nonionic surfactants are C₆ to C₁₂-alkylphenols, alkoxylated with ethylene oxide and/or propylene oxide with a minimum degree of alkoxylation of 2.

Other examples of nonionic surfactants are hydrophilic C₈ to C₁₈-alkyl polyglycosides with 1 to 4 carbohydrate units.

10 Polyethylene glycols may have a molecular weight from 300 to 5000, preferably from 800 to 4000.

Polypropylene glycols may have a molecular weight from 250 to 4000, preferably from 400 to 2500.

Examples of salt-tolerant anionic surfactants are a C₈ to C₁₈-alcoholalkoxy carboxylate salt with a degree of alkoxylation of 1 to 10, or C₆ to C₁₀ aliphatic or aromatic sulphonates.

15 A preferred composition according to the invention comprises from 60 to 90% by weight of component a), from 8 to 38% by weight of water and from 2 to 32% by weight of component c).

The composition according to the invention may comprise some impurities, such as unreacted olefin and inorganic salt. The amounts of them generally do not exceed about 5% by weight each, calculated on the internal olefin sulphonate.

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Examples 1-7

In a falling film reactor of stainless steel, having a length of 6 m and a diameter of 2,54 cm, were introduced internal olefins of different compositions:

25 A. a mixture of internal olefins in the range of from C₁₄ to C₂₀, average molecular weight 230 and a purity of 97%;

B. a mixture of C₁₄ internal olefins and C₁₆ internal olefins, in a weight ratio of 1:2, average molecular weight 214 and a purity of more than 99%; and

30 C. a mixture of C₁₄-, C₁₆- and C₁₈ internal olefins, in a weight ratio of 1:1:1, average molecular weight 222 and a purity of more than 99%.

The sulphur trioxide was prepared by burning sulphur in dry air followed by conversion of the sulphur dioxide to sulphur trioxide over a vanadium pentoxide catalyst at about 450 °C.

The reactor was cooled by flowing water of a temperature of 8 to 11 °C along the outside of the stainless steel reactor tube. The internal olefin mixture (either A, or B or C) flowed along the inner part of the reactor wall as a flowing film in downward direction and was allowed to react cocurrently with the sulphur trioxide, which was introduced at the same time in the top of the reactor, diluted with air. The reaction proceeded continuously.

The reaction mixture coming out of the falling film sulphonation reactor was conducted into a continuous neutralization loop, provided with a combined pump/high sheer mixer and two heat exchangers, and having a volume of about 26 litres; in that continuous neutralization loop the reaction mixture coming from the sulphonation reactor was subjected to neutralization/hydrolysis at 30-45 °C by, at the same time, continuously introducing into the loop streams of i) a concentrated (50% by weight) aqueous sodium hydroxide solution, ii) water, and iii) water plus nonionic surfactant and intimately mixing these streams by means of the combined pump/high sheer mixer and recycling in the neutralization loop. The recycle rate in the loop was about 20 times greater than the intake rate of the reaction mixture coming from the film reactor. The average residence time of the neutralization/hydrolysis mixture in the continuous neutralization loop was dependent on the precise composition of a particular product and of the initial olefin feed rate, and amounted to 40-55 min. The intake rate of the concentrated aqueous sodium hydroxide stream was adjusted on the basis of alkalinity measurements in the end-product (i.e. after the continuous hydrolysis step), which was aimed to be slightly basic. The amounts of water (total) and nonionic surfactant were calculated on the basis of the desired composition. The anionic active matter concentration was actually measured in the end-product (i.e. after the continuous hydrolysis step) during the production.

From the continuous neutralization loop the product mixture exited continuously into a continuous laminar tubular hydrolysis reactor that was heated externally with oil having a temperature of 170-190 °C, the reactor being provided with a preheater section heated with steam at 6 bar, a cooler, and a constant pressure valve; the volume of the hydrolysis reactor was about 40 litres from which the average residence time for each specific product could be estimated. The product flowing out of the constant pressure valve was analysed and, if necessary, adjustments were made in, for instance, the SO₃/olefin ratio, the feed rate

of concentrated sodium hydroxide solution, water, etc. on the basis of these analyses. After these adjustments sufficient time was allowed to obtain representative samples which were then collected and analyzed. The specific reaction conditions and analytical data of the products are given in the Table.

TABLE

| Example | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
|--|-------------------------|--------------|--------------|--------------|--------------|--------------|--------------|
| Internal olefin feed | | | | | | | |
| Internal olefin feed rate, SO ₃ /olefin ratio, | mol olefin/h mol/mol | 71.5 1.06 | 76.5 0.99 | 61.9 0.98 | 59.2 1.02 | 59.2 1.02 | 60.0 1.00 |
| SO ₃ in air, | g volume | 2.5 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 |
| Sulphur burning, | kg/h | 2.5 | 2.5 | 2.0 | 2.0 | 2.0 | 2.0 |
| Neutralization/hydrolysis temp., °C | 35-40 | 35-40 | 40-45 | 37-43 | 37-41 | 37-41 | 38-42 |
| Product data | | | | | | | |
| Anionic active material ¹⁾ | % weight on product | 65.5 | 67.0 | 61.6 | 65.8 | 50.3 | 58.6 |
| Dobanol ^R 91-10 | % weight on product | 3.3 | 6.7 | 6.2 | 6.6 | 2.3 | 2.7 |
| Dobanol ^R 23-9 | % weight on product | - | - | - | - | - | - |
| Free oil ²⁾ | % weight on product | 3.1 | 1.5 | 1.0 | 0.9 | 1.0 | 1.2 |
| Na ₂ SO ₄ ³⁾ | % weight on product | 2.6 | 1.4 | 0.6 | 1.4 | 1.0 | 1.1 |
| Water | | | | | | | |
| Colour "Klett" ⁴⁾ (not bleached) | n.d. | 115 | 120 | 210 | 120 | 110 | 100 |

- 1) two-phase titration using hyamine with a mixed acid indicator
- 2) free oil is the petroleum ether extractable material in an aqueous alcoholic solution of internal olefin sulphonates.
- 5 3) determined according to SMS 1721-76.
- 4) Klett-Summerson photoelectric calorimeter, equipped with a KS No. 42 colour filter, and a 2 x 4 cm glass cell, measured on a 5% active material solution.

10 R = Registered trade mark DOBANOL.

15 Dobanol^R 91-10: a mixture of C₉, C₁₀ and C₁₁ primary alcohols (in a weight ratio of 18:50:32), ethoxylated to an average oxyethylene number of 10.

20 Dobanol^R 23-9: a mixture of C₁₂ and C₁₃ primary alcohols (in a weight ratio of 42:58), ethoxylated to an average oxyethylene number of 9.

25 It is further observed that the compositions given in the Table are all liquid and pourable at 20 °C and can be pumped at that temperature, whilst this holds also for the materials during production in the continuous neutralization loop and the continuous hydrolysis reactor. Reducing during production the amount of nonionic surfactant in Examples 1 and 7 to zero leads to products that can not be pumped through the neutralization/hydrolysis reactor even in spite of the higher temperature in the neutralization loop.

30 **Claims**

1. A concentrated, liquid, pourable composition comprising:
 - a) from 50 to 95% by weight of an alkali or earth alkali metal or ammonium salt or a salt of an organic base of an internal olefin sulphonic acid, having from 8 to 26 carbon atoms, these internal olefin sulphonates having at least 25% by weight of beta-hydroxy sulphonates;
 - b) from 4 to 49% by weight of water; and
 - c) from 1 to 46% by weight of a lower alcohol, a nonionic surfactant, a polyethylene glycol, a polypropylene glycol, a salt-tolerant anionic surfactant, or a mixture thereof.
- 40 2. A composition as claimed in claim 1 wherein the internal olefin sulphonates comprise at least 50% by weight of beta-hydroxy sulphonates.
3. A composition as claimed in claim 1 or 2 wherein the internal olefin sulphonates comprise at least 70% by weight of beta-hydroxy sulphonates.
- 45 4. A composition as claimed in one or more of the claims 1-3, wherein the internal olefin sulphonic acid moiety comprises from 13 to 24 carbon atoms.
5. A composition as claimed in one or more of the claims 1-4 wherein the lower alcohol is methanol, ethanol, propanol, isopropanol, butanol, iso-butanol, tert.butanol, pentanol or hexanol, ethylene glycol, propylene glycol, a linear or branched C₁ to C₆ mono-alkylether of mono- or di-ethylene glycol, a linear or branched C₁ to C₆ mono-alkylether of mono- or di-propylene glycol, or a mixture thereof.
- 55 6. A composition as claimed in one or more of the claims 1-4 wherein the nonionic surfactant is a C₇ to C₁₈-alcohol that is alkoxylated with ethylene oxide and/or propylene oxide with a minimum degree of alkoxylation of 2.

7. A composition as claimed in one or more of the claims 1-4 wherein the nonionic surfactant comprises a C₆ to C₁₂-alkylphenol, alkoxylated with ethylene oxide and/or propylene oxide with a minimum degree of alkoxylation of 2.
- 5 8. A composition as claimed in one or more of the claims 1-4 wherein the nonionic surfactant comprises a hydrophilic C₈ to C₁₈-alkylpolyglycoside with 1 to 4 carbohydrate units.
9. A composition as claimed in one or more of the claims 1-4 comprising polyethylene glycol with a molecular weight from 300 to 5000, preferably from 800 to 4000.
- 10 10. A composition as claimed in one or more of the claims 1-4 comprising polypropylene glycol with a molecular weight from 250 to 4000, preferably from 400 to 2500.
11. A composition as claimed in one or more of the claims 1-4 wherein the salt-tolerant anionic surfactant comprises a C₈ to C₁₈-alcoholalkoxy carboxylate salt with a degree of alkoxylation of 1 to 10, or a C₆ to C₁₀ aliphatic or aromatic sulphonate.

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EUROPEAN SEARCH REPORT

Application Number

| DOCUMENTS CONSIDERED TO BE RELEVANT | | | EP 91202510.3 |
|--|--|---|---|
| Category | Citation of document with indication, where appropriate, of relevant passages | Relevant to claim | CLASSIFICATION OF THE APPLICATION (Int. Cl.5) |
| X | <p><u>US - A - 4 715 991</u> (HIRAKOUCHI et al.) * Column 2, line 9 - column 3, line 23; column 4, lines 5-10; tables *</p> <p>---</p> | 1, 2, 3, 4, 5, 6, 7, 8, 11 | C 11 D 1/14 |
| A | <p><u>US - A - 4 309 317</u> (SEKIGUCHI et al.) * Example 1; claim 1 *</p> <p>----</p> | 1 | |
| TECHNICAL FIELDS SEARCHED (Int. Cl.5) | | | |
| C 11 D | | | |
| <p>The present search report has been drawn up for all claims</p> | | | |
| Place of search | Date of completion of the search | | Examiner |
| VIENNA | 28-11-1991 | | SEIRAFI |
| CATEGORY OF CITED DOCUMENTS | | <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p> | |
| <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> | | | |