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54 **Process for preparing detergent compositions.**

57 A process for preparing a liquid surfactant composition comprising an anionic surfactant and a nonionic surfactant and having a relatively low water content, whereby essentially equimolar amounts of a neutralizing agent and a liquid acid precursor of the anionic surfactant are blended simultaneously in the presence of the nonionic surfactant. The process is especially suitable for preparing mixtures of nonionic and primary alkyl sulphate.

TECHNICAL FIELD

The present invention relates to a process for preparing liquid detergent compositions comprising an anionic surfactant and a nonionic surfactant. More in particular, it relates to a process for the continuous preparation of a liquid detergent composition comprising an anionic and a nonionic surfactant, and having a high surfactant content.

BACKGROUND AND PRIOR ART

EP-A-265 203 (Unilever) discloses liquid surfactant compositions which are mobile at 20 to 80 °C and contain a sodium or potassium salt of an alkylbenzene sulphonate or alkyl sulphate in an amount not exceeding 80% by weight; an ethoxylated nonionic surfactant in an amount not exceeding 80% by weight; and water in an amount not exceeding 10% by weight. Such liquid surfactant composition may be used as a concentrated liquid detergent, for example as a dishwashing liquid. Alternatively, the liquid surfactant composition may be sprayed onto a solid particulate absorbent material, for instance a porous spray-dried base powder having a low bulk density and containing no or little actives, to form a detergent base powder having an increased bulk density.

EP-A-265 203 describes two methods for preparing such concentrated liquid surfactant compositions. According to the first method, liquid nonionic surfactant is gradually added to a neutralized alkylbenzene paste (neutralized salt) which will typically have an active matter content of about 50% by weight. The resulting viscous mixture containing more than 10% water, is then heated to evaporate a sufficient amount of water such that the final water content will fall below 10%.

According to the second method, alkylbenzene sulphonic acid is mixed with nonionic surfactant, and the mixture is subsequently treated with a sufficient amount of concentrated aqueous sodium hydroxide or potassium hydroxide to effect partial or complete neutralization.

By means of the above processes good results may be obtained in the preparation of liquid detergent compositions. However, the first method involves the evaporation of water and is therefore time consuming and energy consuming. A drawback of the second method is that prolonged interaction between a nonionic surfactant and an acid substance such as an acid anionic surfactant precursor may cause chemical degradation of the nonionic whereby dioxane may be formed.

It is therefore an object of the present invention to provide an improved process for preparing a liquid surfactant composition comprising an anionic surfactant and a nonionic surfactant and having a relatively low water content.

We have now found that an improved process for the preparation of the afore mentioned kind of liquid surfactant composition may be obtained according to the present invention. In this process, essentially equimolar amounts of a liquid acid anionic surfactant precursor and a neutralizing agent are blended simultaneously in the presence of nonionic surfactant.

DEFINITION OF THE INVENTION

The present invention provides a process for preparing a liquid surfactant composition comprising an anionic surfactant and a nonionic surfactant and having a relatively low water content, whereby essentially equimolar amounts of a neutralizing agent and a liquid acid precursor of the anionic surfactant are blended simultaneously in the presence of the nonionic surfactant. Preferably, the process is carried out continuously, more preferably in a loop reactor.

DETAILED DESCRIPTION OF THE INVENTION

The principle of the process of the invention is to neutralize the acid corresponding to the anionic surfactant with a neutralizing agent of a strength such as to lead to the desired low level of water in the final product by adding these two materials to a fluid which comprises the nonionic and which acts as a solvent or diluent for the neutralized anionic surfactant.

The liquid acid corresponding to the anionic surfactant or acid anionic surfactant precursor is usually a stable organic compound which is commercially available. An example is Dobanic 113, C₁₂-C₁₅ alkylbenzene sulphonic acid, which can be obtained from Shell.

However, the acid anionic surfactant precursors of primary alkyl sulphates are believed to be less stable and are therefore advantageously prepared directly before the neutralization reaction by sulphating the corresponding alcohols.

The neutralizing agent can be in principle any suitable alkaline substance, preferably in a concentrated aqueous solution. Concentrated aqueous solutions of an alkali metal hydroxide, such as a 50% by weight solution of sodium hydroxide in water are the preferred neutralizing agents. Alternatively, concentrated alkaline silicate solutions could be used.

5 The process of the invention for preparing the surfactant mixtures may be carried out batch-wise, for instance by adding equimolar amounts of the anionic precursor and the neutralizing agent to a reaction vessel containing the required amount of nonionic surfactant under continuous stirring and at a controlled temperature.

10 Preferably, however, the process is carried out continuously, by feeding the anionic surfactant precursor, the nonionic surfactant and the neutralizing agent continuously and in the required amounts into a reactor and collecting the formed liquid surfactant composition after neutralization has taken place.

According to an especially preferred embodiment of the invention, the process is carried out continuously in a loop reactor. Such loop reactors are known in the art and have, as far as we are aware, only been used for carrying out the neutralization of acid anionic surfactant precursors with aqueous sodium hydroxide.

15 The liquid surfactant compositions prepared according to the present invention usually correspond to the liquid surfactant compositions described in the European patent application 265,203, but also liquid compositions having a higher water content may be prepared. The water content of the surfactant mixtures prepared according to the present invention is less than 25% by weight, preferably less than 15% by weight. A water content of less than 10% by weight is especially preferred.

20 The prepared liquid surfactant compositions comprise 1 to 80%, preferably 15 to 80% by weight of one or more anionic surfactants, 10 to 99%, preferably 10 to 85% by weight of one or more nonionic surfactants and as little water as possible, in order to keep the composition mobile in the temperature range of 20-95 °C. Most preferred ranges for the surfactants are 30 to 70% by weight for the anionic and 30 to 70% by weight for the nonionic.

25 The anionic surfactant component in the composition may be a sodium or potassium salt of a liquid acid surfactant precursor. It may be an alkyl sulphate, an alkylbenzene sulphonate, an alkyl ether sulphate or an fatty acid ether sulphate. Particularly suitable are sodium alkyl sulphates wherein the alkyl group possesses 10 to 20, preferably 12 to 18 carbon atoms, and sodium alkyl benzene sulphonates wherein the alkyl group possesses 10 to 18 carbon atoms. Mixtures of these anionic surfactants may also be used.

30 The nonionic surfactant may be any suitable nonionic surfactant that is liquid or readily liquefiable at temperatures up to about 80 °C. Suitable types of nonionic surfactants for this purpose are the ethoxylated fatty alcohols, for example a C₁₂-C₁₅ aliphatic alcohol ethoxylated with 2 to 10 moles of ethylene oxide per mole of alcohol. Examples of preferred nonionics are the C₁₃-C₁₅ fatty alcohols condensed with 3 or 7 ethoxy groups, such as those commercially available from ICI as Synperonics A3 or A7 (Trade Marks).

35 The weight ratio between the anionic surfactant and the nonionic surfactant in the obtained surfactant mixture is preferably in the range of 0.125:1 to 4:1. However, compositions having a high an anionic to nonionic ratio of more than about 1.5:1 were found to be in general more viscous and are therefore less preferred. The especially preferred compositions prepared according to the present process have an anionic to nonionic ratio of from 0.2:1 to 2:1.

40 The liquid surfactant compositions prepared according to the process of the present invention may additionally contain free fatty acid. Thus they may additionally comprise 0.5 to 20%, preferably 2 to 15%, more preferably 2 to 7% by weight of a fatty acid having 8 to 22 carbon atoms. It is preferred if the fatty acid possesses 12 to 20 carbon atoms, and more in particular 16 to 18 carbon atoms.

45 The liquid surfactant compositions prepared according to the process of the present invention may be favourably applied in a process for making high bulk density granular detergent compositions having a high active detergent level, as disclosed by EP-A- 367 339.

The invention is further illustrated by the following non-limiting Examples, in which parts and percentages are by weight, unless otherwise indicated.

50 The following abbreviations are used :

ABS : C₁₂-C₁₅ alkylbenzene sulphonic acid, Dobanic 113 ex Shell

PAS : Primary alkyl sulphate, obtained by sulphating a C₁₂-C₁₃ or C₁₂-C₁₅ alcohol (Lial 123 or 125 respectively, ex Enichem)

LES : Lauryl ether sulphate

55 NI : Nonionic surfactant (ethoxylated C₁₃-C₁₅ fatty alcohol)

EXAMPLES 1-3

The following mobile liquid Alkylbenzene sulphonate/Nonionic surfactant mixtures were prepared in a conventional neutralization loop reactor. The reactor is essentially in the form of a closed loop and comprises an in-line mixer. Down stream is a heat exchanging facility and before the mixer there are three inlets which are used for the liquid acid anionic precursor, the nonionic and the aqueous sodium hydroxide. Further up stream an outlet is provided through which the neutralized surfactant composition may be bled off, the majority of the product being recirculated into the loop.

For start up the loop was filled with nonionic surfactant, a blend of Synperonic A3 and Synperonic A7 in a ratio of 4 : 3. Subsequently, the nonionic surfactant mixture, aqueous 48% by weight sodium hydroxide solution and liquid anionic acid precursor (Dobanic 113) were dosed in continuously, whereby the amount of sodium hydroxide was chosen to be just sufficient to neutralize the acid. In order to obtain different anionic to nonionic ratios, the nonionic feed rate was set at three different levels. The feed rates of the starting materials and the calculated compositions of the obtained surfactant blend are given below. The coolant temperature was 50 °C.

Example	1	2	3
Anionic acid Feed Rate (kg/hr)	10	10	10
NaOH (48%) Feed Rate (kg/hr)	2.59	2.59	2.59
NI Feed Rate (kg/hr)	21	11.5	5.25
% Anionic detergent	30.7	44.0	54.7
% Nonionic detergent	63.6	48.3	35.7
% H ₂ O	5.7	7.7	10
Ratio Anionic/Nonionic	0.5	1	2

The surfactant compositions of Examples 1 and 2 were easily handleable in the loop and were mobile liquids at room temperature. The composition of Example 3 was more viscous and was therefore more difficult to handle.

EXAMPLES 4-6

The Examples 1-3 were repeated, except that a primary alkyl sulphate (PAS) was used as anionic surfactant and Synperonic A7 as the only nonionic surfactant. The PAS acid precursor was prepared by sulphating Lial 123, a C₁₂-C₁₃ alkanol with SO₃ in a falling film reactor (FFR). The PAS acid was fed into the loop reactor directly after it was prepared. The feed rates of the starting materials and the calculated composition of the obtained surfactant blend are given below. The coolant temperature was 50 °C and the pH of the final composition was between 11 and 14.

Example	4	5	6
Lial 123 to FFR Feed Rate (kg/hr)	10	10	10
SO ₃ (4% in air) to FFR Feed Rate (kg/hr)	4.9	4.9	4.9
NaOH (48%) Feed Rate (kg/hr)	4.2	4.2	4.2
NI Feed Rate (kg/hr)	29	14.5	7.25
% Anionic detergent	31.5	45.4	58.3
% Nonionic detergent	61.3	44.2	28.4
% H ₂ O	6.5	9.4	12.1
Ratio Anionic/Nonionic	0.5	1	2

The surfactant compositions of Examples 4 and 5 were easily handleable in the loop and were mobile liquids at room temperature. The composition of Example 6 was more viscous and was therefore more difficult to handle.

EXAMPLES 7-8

The Examples 4-6 were repeated, under slightly different conditions and using Lial 125 (a C₁₂-C₁₅ alcohol) as starting alcohol. The feed rates of the starting materials and the composition of the obtained surfactant blend are given below.

Example	7	8
Lial 125 to FFR Feed Rate (kg/hr)	10	10
SO ₃ (4% in air) to FFR Feed Rate (kg/hr)	4.42	4.42
NaOH (48%) Feed Rate (kg/hr)	4.17	4.17
NI Feed Rate (kg/hr)	25.2	12.6
% Anionic detergent (analyzed)	28.4	41.1
% Nonionic detergent (calculated)	58.4	41.2
% H ₂ O	7.1	10.0
Ratio Anionic/Nonionic	0.5	1

EXAMPLE 9

The Examples 4-6 were repeated, except that lauryl ether sulphate (LES) was used as anionic surfactant. The LES acid precursor was prepared by sulphating Synperonic A3 nonionic in a falling film reactor (FFR). Synperonic A7 was used as the only nonionic. The feed rate of the starting materials and the composition of the obtained surfactant blend (as analyzed) are given below. The coolant temperature was 50° C and the pH of the final composition was between 11 and 14.

Example	9
Synperonic A3 to FFR Feed Rate (kg/hr)	10
SO ₃ (4% in air) to FFR Feed Rate (kg/hr)	4.54
NaOH (48%) Feed Rate (kg/hr)	4.1
NI Feed Rate (kg/hr)	29
% Anionic detergent	30.4
% Nonionic detergent	62.5
% H ₂ O	7.1
Ratio Anionic/Nonionic	0.5

The obtained surfactant composition was easily handleable in the loop and was fluid at room temperature.

Claims

1. A process for preparing a liquid surfactant composition comprising an anionic surfactant and a nonionic surfactant and having a relatively low water content, whereby essentially equimolar amounts of a neutralizing agent and a liquid acid precursor of the anionic surfactant are blended simultaneously in the presence of the nonionic surfactant.
2. A process according to Claim 1, wherein the process is carried out continuously.
3. A process according to any one of the preceding Claims, wherein the process is carried out continuously in a loop reactor.
4. A process according to any one of the preceding Claims, wherein the anionic surfactant is an alkylbenzene sulphonate.
5. A process according to any one of Claims 1-3, wherein the anionic surfactant is a primary alkyl sulphate.
6. A process according to any one of the preceding Claims, wherein the nonionic surfactant is an ethoxylated fatty alcohol.
7. A process according to Claim 6, wherein the nonionic surfactant is a C₁₂-C₁₅ fatty alcohol, ethoxylated with 3 to 7 ethoxy groups.

8. A process according to any one of the preceding Claims, wherein the neutralizing agent is concentrated aqueous alkali metal hydroxide.

5 9. A process according to any one of the preceding Claims, wherein the obtained surfactant composition has a water content of less than 25% by weight.

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European Patent
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EUROPEAN SEARCH REPORT

Application Number

EP 92 20 0908

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	PATENT ABSTRACTS OF JAPAN vol. 13, no. 238 (C-603)5 June 1989 & JP-A-1 047 755 (DAI ICHI KOGYO SEIYAKU CO LTD) 22 February 1989 * abstract *	1, 4-6, 8, 9	C11D11/04 C07C303/32
X	EP-A-0 364 880 (SUNSTAR INC.) * page 4, line 2 - line 10; claim 2 *	1, 4-6, 8, 9	
A	DE-A-2 232 341 (ORTMANN & HERBST GMBH) * claims *	1-3	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C110 C07C C11C
The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 02 JULY 1992	Examiner PELLI-WABLAT B.
CATEGORY OF CITED DOCUMENTS 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 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			