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(54) Laundry detergent particles

(57) The present invention provides lenticular or disc detergent particle.

EP 2 441 821 A1

Description

Field of Invention

[0001] The present invention relates to large laundry detergent particles.

Background of Invention

[0002] CA 23000786 discloses that by adding a co surfactant to MES it is possible to lower the Kraft point of the MES and hence provide better dissolution of a MES detergent in a cold water wash medium.

[0003] WO2009/050026 (C4569) discloses that by adding a co surfactant to MES it is possible to lower the Kraft point of the MES and hence provide better dissolution of a MES detergent in a cold water wash medium.

[0004] WO9932599 describes a method of manufacturing laundry detergent particles, being an extrusion method in which a builder and surfactant, the latter comprising as a major component a sulphated or sulphonated anionic surfactant, are fed into an extruder, mechanically worked at a temperature of at least 40°C, preferably at least 60°C, and extruded through an extrusion head having a multiplicity of extrusion apertures. In most examples, the surfactant is fed to the extruder along with builder in a weight ratio of more than 1 part builder to 2 parts surfactant. The extrudate apparently required further drying. In Example 6, PAS paste was dried and extruded. Such PAS noodles are well known in the prior art. The noodles are typically cylindrical in shape and their length exceeds their diameter, as described in example 2.

[0005] US 7,022,660 discloses a process for the preparation of a detergent particle having a coating.

[0006] In a further aspect, the present invention provides a coated detergent particle that is a concentrated formulation with more surfactant than inorganic solid. Only by having the coating encasing the surfactant which is soft can one have such a particulate concentrate where the unit dose required for a wash is reduced. Adding solvent to the core would result by converting the particle into a liquid formulation. On the other hand, having a greater amount of inorganic solid would result in a less concentrated formulation; a high inorganic content would take one back to conventional low surfactant concentration granular powder. The coated detergent particle of the present invention sits in the middle of the two conventional (liquid and granular) formats.

Summary of the Invention

[0007] Alkyl ester fatty acids tend to be crystalline surfactants. The addition of co-surfactant to alkyl ester fatty acid, such as an anionic and/or non-ionic to alkyl ester fatty acids results in the mixture being an amorphous form rather than crystalline; the amorphous form is sticker than the crystalline form which results is processing negatives and caking on storage.

[0008] We have found that the negatives associated with a mixture of alkyl ester fatty acid and co-surfactant may be ameliorated by processing the mixture to a large particle having an inorganic salt coating.

[0009] In one aspect the present invention provides a coated detergent particle having perpendicular dimensions x, y and z, wherein x is from 1 to 2 mm, y is from 2 to 8mm (preferably 3 to 8 mm), and z is from 2 to 8 mm (preferably 3 to 8 mm), wherein the particle comprises:

(i) from 40 to 90 wt % of a surfactant mixture, preferably 50 to 90 wt%, the surfactant mixture being alkyl ester fatty acid and a co-surfactant, wherein the ratio of alkyl ester fatty acid:co-surfactant is in the range from 60:40 to 90:10, more preferably 70:30 to 85:15 (optimally 80:20);
(ii) from 1 to 40 wt %, preferably 20 to 40 wt %, water soluble inorganic salts; and,
(iii) from 0 to 3 wt%, preferably 0.001 to 3 wt % of a perfume,
wherein the inorganic salts are present on the detergent particle as a coating and the surfactant mixture present as a core.

[0010] Reference to alkyl ester fatty acid as found herein is to the sodium salts thereof.

[0011] Unless otherwise stated all wt % refer to the total percentage in the particle as dry weights.

[0012] In a further aspect, the present invention provides a coated detergent particle that is a concentrated formulation with more surfactant than inorganic solid. Only by having the coating encasing the surfactant which is soft can one have such a particulate concentrate where the unit dose required for a wash is reduced. Adding solvent to the core would result by converting the particle into a liquid formulation. On the other hand, having a greater amount of inorganic solid would result in a less concentrated formulation; a high inorganic content would take one back to conventional low surfactant concentration granular powder. The coated detergent particle of the present invention sits in the middle of the two conventional (liquid and granular) formats.

Detailed Description of the Invention

[0013] The co-surfactant is other than MES.

SHAPE

[0014] Preferably the coated laundry detergent particle is curved.

[0015] The coated laundry detergent particle may be lenticular (shaped like a whole dried lentil), an oblate ellipsoid, where z and y are the equatorial diameters and x is the polar diameter; preferably y = z.

[0016] The coated laundry detergent particle may be shaped as a disc.

[0017] Preferably the coated laundry detergent particle does not have hole; that is to say, the coated laundry detergent particle does not have a conduit passing there though that passes through the core, i.e., the coated detergent particle has a topologic genus of zero.

LAS

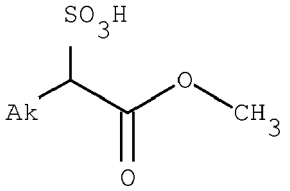
[0018] The alkyl benzene sulphonate may be branched for example tetrapropylenebenzenesulphonate or linear for example linear alkyl benzene sulphonate. Preferably the alkyl benzene sulphonate is linear alkyl benzene sulphonate.

ALKYL ESTER FATTY ACID

[0019] The sulphonated alkyl ester may be pure alkyl ester sulphonate or a blend of a mono-salt of a sulphonated alkyl ester of a fatty acid having from 16 to 26 carbon atoms where the alkyl portion forming the ester is a straight or branched chain alkyl of 1 to 6 carbon atoms and a disalt of a sulphonated fatty acid. The ratio of monosalt to disalt being at least 2:1 and up to about 25:1. The sulphonated alkyl esters used are typically prepared by sulphonating an alkyl ester of a fatty acid with a sulphonating agent such as SO_3. When prepared in this way the sulphonated alkyl esters normally contain a minor amount of the disalt of the sulphonated fatty acid which results from hydrolysis of the ester. Preferred sulphonated alkyl esters contain less than about 10% by weight of the disalt of the corresponding sulphonated fatty acid.

[0020] The sulphonated alkyl esters, include linear esters of C16 to C26 carboxylic acid which are sulphonated with gaseous SO_3 according to the Journal of American oil Chemists Society 52 (1975) pp 323-329. Suitable starting materials would include natural fatty substances as derived from tallow, palm oil, coconut etc

[0021] The preferred alkyl ester sulphonate or fatty acid sulphonate surfactants comprise alkyl sulphonate surfactants of the type methyl ester fatty acid sulphonate (MES), having the formula:



where Ak = predominantly linear C_8 - C_{20} alkyl chain, preferably C_{16} - C_{18}.

[0022] The IUPAC name for e.g. a so-called C16 MES is Hexadecanoic acid, 2-sulfo-, 1-methylester, sodium salt (C17H33NaO5S).

[0023] Methyl ester sulphonate can be obtained by sulphonation of various renewable oleo-based methyl ester feedstocks derived from e.g. coconut (C12-14), palm kernel (C8-18), palm stearin (C16-18) or tallow (C16-18). Besides the renewable origin, MES is of special interest due to good biodegradability, detergency and calcium hardness tolerance.

[0024] Methods of preparing α-sulfofatty acid esters are known to skilled artisan. (See, e.g., US-A-5 587 500; US-A-5 384 422; US-A-5 382 677; US-A-5 329 030; US-A-4 816 188; and US-A- 4 671 900; and The Journal of American Oil Chemists Society 52:323-29 (1975) the disclosures of which are incorporated herein by reference). α-Sulfofatty acid esters can be prepared from a variety of sources, including beef tallow, palm kernel oil, palm kernel (olefin) oil, palm kernel (stearin) oil, coconut oil, soybean oil, canola oil, cohune oil, coco butter, palm oil, white grease, cottonseed oil, corn oil, rape seed oil, soybean oil, yellow grease, mixtures thereof or fractions thereof. Suitable fatty acids to make α-sulfofatty acid esters include, but are not limited to, caprylic (C_8), capric ((C_{10}), lauric (C_{12}), myristic (C_{14}), myristoleic (C_{14}), palmitic (C_{16}), palmitoleic (C_{16}), stearic (C_{18}), oleic (C_{18}), linoleic (C_{18}), linolenic (C_{18}), ricinoleic (C_{18}), arachidic

(C_{20}), gadolic (C_{20}), behenic (C_{22}), and erucic (C_{22}) fatty acids. α-Sulfofatty acid esters prepared from one or more of these sources are within the scope of the present invention.

[0025] Samples of alkyl ester fatty acid sulphonate surfactant prepared in dry powder formed via above mentioned production processes typically contain about 75-85 % by weight of the desired surfactant on surfactant.

Water soluble inorganic salts

[0026] The water soluble inorganic salts are preferably selected from sodium carbonate, sodium chloride, sodium silicate and sodium sulphate, or mixtures thereof, most preferably 70 to 100 wt % sodium carbonate on water soluble inorganic salt. The water soluble inorganic salt is present as a coating on the particle. The water soluble inorganic salt is preferably present at a level that reduces the stickiness of the laundry detergent particle to a point where the particles are free flowing.

[0027] It will be appreciated by those skilled in the art that multiple layered coatings, of the same or different coating materials, could be applied, but a single coating layer is preferred, for simplicity of operation, and to maximise the thickness of the coating. The amount of coating should lay in the range 1 to 40 wt % of the particle, preferably 20 to 40 wt %, even more preferably 25 to 35 wt % for the best results in terms of anti-caking properties of the detergent particles.

[0028] The coating is applied to the surface of the surfactant core, by crystallisation from an aqueous solution of the water soluble inorganic salt. The aqueous solution preferably contains greater than 50g/L, more preferably 200 g/L of the salt. An aqueous spray-on of the coating solution in a fluidised bed has been found to give good results and may also generate a slight rounding of the detergent particles during the fluidisation process. Drying and/or cooling may be needed to finish the process.

[0029] By coating the large detergent particles of the current invention the thickness of coating obtainable by use of a coating level of say 5 wt% is much greater than would be achieved on typically sized detergent granules (0.5-2mm diameter sphere).

[0030] For optimum dissolution properties, this surface area to volume ratio must be greater than 3 mm^{-1}. However, the coating thickness is inversely proportional to this coefficient and hence for the coating the ratio "Surface area of coated particle" divided by "Volume of coated particle" should be less than 15 mm^{-1}.

[0031] It is preferred that the coated detergent particle has a core to shell ratio of from 4 to 1:1, most preferably 3 to 1.5:1; the optimal ratio of core to shell is 2:1.

EXPERIMENTAL

[0032] A range of blends of these two surfactants, (a MES/LAS ratio from 90/10 to 70/30) have been manufactured at lab scale to simulate this process. These powders have been stored at Relative Humidities from 15 to 75% to achieve a range of moisture contents. The resulting materials have been assessed for acceptability to mill using a Moulinette and dissolution by t(90) value; these results are shown in Tables 1-3 below.

Table 1 - 90:10 MES/LAS

Storage RH (%)	15	33	53	75
Total weight (g)		36.7	38.2	41.16
Actual water activity (aw)				
Actual ERH (%)		26.4	35.1	45.3
Moisture content (%)		2.79	3.08	8.42
Millable at 20ºC		Yes	Yes	Yes
t(90) (355-500um)		13.2	17.7	52.5*
Hardness [Pa]		132.37	73.67	20.31

Table 2 - 80:20 MES/LAS

Storage RH (%)	15	33	53	75
Total weight (g)	NM	NM	NM	45.8
Actual water activity (aw)	0.256	0.44	0.56	0.76

(continued)

Storage RH (%)	15	33	53	75
Actual ERH (%)	25.6	44	56	76
Moisture content (%)	5.28	5.1	8.2	10.6
Millable at 20°C	Yes	No	No	No
t(90) (355-500um)	15	16	14	29*
Hardness [Pa]	8.64	1.99	1.87	0.45

Table 3 - 70:30 MES/LAS

Storage RH (%)	15	33	53	75
Total weight (g)	42.4	41.71	42.4	44.61
Actual water activity (aw)	0.257	0.544	0.632	0.739
Actual ERH (%)	25.7	54.4	63.2	73.9
Moisture content (%)	5.63	6.67	7.55	11.26
Millable at 20º C	No	No	No	No
t(90) (355-500um)	14		20*	21*
Hardness [Pa]	2.14		0.62	0.14

Note: Sample t(90) results marked with an asterisk (*) come from materials which clumped after milling. The actual particle size of the sample prepared for the t(90) may be larger than 355-500 microns.

[0033] The results for the MES/LAS blends indicate that all have acceptable dissolution properties. However as the level of LAS is increased, there is a tendency to become more adhesive as shown by the inability to mill the material. As expected, the hardness also decreases with increasing LAS content and moisture content. These results indicate that it should be possible to manufacture a maximum ratio of 80/20 MES/LAS with a target moisture content of up to 6%.

Pilot Plant

[0034] Based on the lab-based findings a plant trial was carried out using an 80/20 ratio of MES/LAS. The MES/LAS paste was dried as described previously,(2) to a range of moisture contents and material assessed for milling and dissolution behaviour.

[0035] The MES/LAS mixtures were dried to moisture contents in the range 1.66% to 2.62%. All of these materials were found to be millable in agreement with the earlier lab-based results. Examination of dissolution behaviour 'as-made' and after storage at 52%RH also found all to be acceptable, with T90 values increasing with MES moisture content. These results seem to confirm the earlier lab-based study however further storage work at elevated temperature,(26°C) such as would be regularly experienced in Asia found a marked tendency for the material to soft cake even in sealed containers. This behaviour means that an 80/20 MES/LAS blend is not acceptable for incorporation in laundry detergent powders.

EXPERIMENTAL

Example 1

[0036] Surfactant raw materials were mixed together to give a 67wt% active paste comprising 80 parts MES and 20 parts LAS.

[0037] Raw Materials used were:

LABSA
Caustic (48% Solution)
MES ex Huish

[0038] The paste was pre-heated to the feed temperature and fed to the top of a wiped film evaporator to reduce the moisture content and produce a solid intimate surfactant blend, which passed the calcium tolerance test. The conditions used to produce this MES/LAS blend are given in Table 1.

Table 1

	Jacket Vessel Temp.	81 °C
Feed	Nominal Throughput	55 kg/hr
	Temperature	70 °C
	Density	1.1 kg/l
Product	Moisture(KF*)	1.66 %
*analysed by Karl Fischer method		

[0039] On exit from the base of the wiped film evaporator, the dried surfactant blend dropped onto a chill roll, where it was cooled to less than 30°C.

[0040] After leaving the chill roll, the cooled dried surfactant blend particles were milled using a hammer mill. The resulting milled material is hygroscopic and so it was stored in sealed containers.

[0041] The cooled dried milled composition was fed to a twin-screw co-rotating extruder fitted with a shaped orifice plate and cutter blade.

[0042] The average particle diameter and thickness of samples of the extruded particles were found to be 4.8 mm and 1.2 mm respectively. The standard deviation was acceptably low.

[0043] The particles were then coated using a Strea 1 fluid bed. The coating was added as an aqueous solution and coating completed under conditions given in Table. Coating wt% is based on weight of the coated particle.

Table 3

Target coating Level	5wt%	10wt%	15wt%
Mass Solid [kg]	1.25	1.25	1.25
Coating Solution	Sodium Carbonate (25%)	Sodium Carbonate (25%)	Sodium Carbonate (25%)
	Dye (0.1%)	Dye (0.1%)	Dye (0.1%)
Mass Coating Solution [kg]	0.27	0.56	0.89
Air Inlet Temperature [°C]	70	70	70
Air Outlet Temperature [°C]	42	40	41
Coating Feed Rate [g/min]	14	15	15
Coating Feed temperature [°C]	38	41	40

[0044] As can be seen from Table 3 the samples have different coating levels. These samples and additional samples made using the same process were then equilibrated at 48 and 65% relative humidity and 28C.

[0045] At the end of the storage the flow properties were assessed.

Sample 1 Fail - clumped on storage at 48 and 65% RH
Sample 2 Fail - clumped on storage at 48 and 65% RH
Sample 3 Pass - free flowing on storage at 48 and 65% RH

Claims

1. A coated detergent particle having perpendicular dimensions x, y and z, wherein x is from 1 to 2 mm, y is from 2 to 8mm, and z is from 2 to 8 mm, wherein the particle comprises:

(i) from 40 to 90 wt % of a surfactant mixture, the surfactant mixture being alkyl ester fatty acid and a co-surfactant, wherein the ratio of alkyl ester fatty acid:co-surfactant is in the range from 60:40 to 90:10; and,
(ii) from 1 to 40 wt % water soluble inorganic salts; and,
(iii) from 0 to 3 wt % of a perfume,
wherein the inorganic salts are present on the detergent particle as a coating and the surfactant mixture present as a core.

2. A coated detergent particle according to claim 1, wherein the ratio of alkyl ester fatty acid:co-surfactant is 70:30 to 85:15.

3. A coated detergent particle according to claim 1 or 2, wherein the co-surfactant is LAS.

4. A coated detergent particle according to any preceding claim, wherein the alkyl ester fatty acid is MES.

5. A coated detergent particle according to any preceding claim, wherein the alkyl ester fatty acid is C16/C18 MES.

6. A coated detergent particle according to any one of the preceding claims, wherein the inorganic salts act as a builder.

7. A coated detergent particle according to claim 6, wherein the inorganic salts comprises sodium carbonate.

8. A coated detergent particle according to any one of the preceding claims, wherein the coated detergent particle comprises from 15 to 85 wt % anionic surfactant on surfactant and from 5 to 75 wt % non-ionic surfactant on surfactant.

9. A coated detergent particle according to any one of claims 1 to 7, wherein the coated detergent particle comprises 15 to 100 wt % anionic surfactant on surfactant of which 20 to 30 wt % is sodium lauryl ether sulphate.

10. A coated detergent particle according to any one of the preceding claims, wherein the anionic surfactant is selected from alkyl benzene sulphonates; alkyl ether sulphates; alkyl sulphates.

11. A coated detergent particle according to any one of the preceding claims, wherein the coated detergent particle comprises 20 to 40 wt % of inorganic builder salts as a coating.

12. A coated detergent particle according to claim 11, wherein the coated detergent particle comprises 25 to 35 wt % of inorganic builder salts as a coating.

13. A coated detergent particle according to any one of the preceding claims, wherein the particle comprises from 0 to 15 wt % water.

14. A coated detergent particle according to claim 13, wherein the particle comprises from 1 to 5 wt % water.

15. A coated detergent particle according to any one of the preceding claims, wherein the coated detergent particle comprises from 10 to 100 wt % of a detergent formulation in a package.

16. A coated detergent particle according to claim 15, wherein the coated detergent particle comprises from 50 to 100 wt % of a detergent formulation in a package.

17. A coated detergent particle according to claim 16, wherein the coated detergent particle comprises from 80 to 100 wt % of a detergent formulation in a package.

18. A coated detergent particle according to claim 17, wherein the coated detergent particle comprises from 90 to 100 wt % of a detergent formulation in a package.

19. A coated detergent particle according to any one of the preceding claims, wherein at least 90 to 100 % of the coated detergent particles in the in the x, y and z dimensions are within a 20 % variable from the largest to the smallest coated detergent particle.

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EUROPEAN SEARCH REPORT

Application Number
EP 10 18 7518

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Y,D	WO 2009/050026 A2 (UNILEVER NV [NL]; UNILEVER PLC [GB]; UNILEVER HINDUSTAN [IN]; ANANTHAS) 23 April 2009 (2009-04-23) * page 1, lines 1-7; claims 1-12; tables 1-3 * * page 1, line 28 - page 2, line 3 * * page 4, line 1 - page 9, line 5 *	1-19	TECHNICAL FIELDS SEARCHED (IPC) C11D
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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Munich	24 March 2011	Klier, Erich

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EUROPEAN SEARCH REPORT

Application Number
EP 10 18 7518

DOCUMENTS CONSIDERED TO BE RELEVANT

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TECHNICAL FIELDS SEARCHED (IPC)

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Munich	24 March 2011	Klier, Erich

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

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