



(11) **EP 2 167 624 B1**

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention
of the grant of the patent:
01.12.2010 Bulletin 2010/48

(51) Int Cl.:
C11D 1/22 ^(2006.01) **C11D 3/10** ^(2006.01)
C11D 3/12 ^(2006.01) **C11D 3/20** ^(2006.01)

(21) Application number: **08761382.4**

(86) International application number:
PCT/EP2008/058137

(22) Date of filing: **26.06.2008**

(87) International publication number:
WO 2009/010375 (22.01.2009 Gazette 2009/04)

(54) **A SOLID DETERGENT COMPOSITION**

FESTES WASCHMITTEL

COMPOSITION DÉTERGENTE SOLIDE

(84) Designated Contracting States:
**AT BE BG CH CY CZ DE DK EE ES FI FR GB GR
HR HU IE IS IT LI LT LU LV MC MT NL NO PL PT
RO SE SI SK TR**

(30) Priority: **16.07.2007 IN MU13542007**
19.11.2007 EP 07120954

(43) Date of publication of application:
31.03.2010 Bulletin 2010/13

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US-A- 3 703 772 US-A- 4 235 758

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DescriptionTECHNICAL FIELD

5 **[0001]** The invention relates to a solid detergent composition comprising a carbonate builder system.

BACKGROUND AND RELATED ART

10 **[0002]** Soaps, which are alkali metal salts of fatty acids, have traditionally been used for the purpose of personal washing. Soaps have also been used for washing laundry. When washing laundry with soaps, the efficiency of washing is lower when washed in hard water. Hard water refers to water having high levels of dissolved Calcium and Magnesium salts. The dissolved Calcium and Magnesium ions react very quickly with the alkali metal cation (sodium or potassium) of the soap, leading to formation of Calcium soap which is insoluble in water and therefore leading to poor detergency. With the advent of synthetic detergents which are alkali metal salts of long chain acids of petroleum origin, the same
15 problem persists. Most popular synthetic detergents include linear alkyl benzene sulphonates, alpha olefin sulphonates, and primary alkyl sulphates which belong to the class of anionic surfactants. Surfactants of the non-ionic, cationic, amphoteric and zwitterionic character are also known. Cleaning performance of most synthetic surfactants is also affected by the washing in hard water.

20 **[0003]** Compounds that react preferentially with the dissolved Calcium and Magnesium ions present in hard water, thereby maintaining the desired high concentration of the detergent in its active form, have been used in detergent compositions. Such compounds or mixture of compounds are known as detergency builders. Commonly known detergency builders are alkali metal carbonates, silicates, phosphates and structured compounds like Zeolites. Alkali metal carbonates like Sodium Carbonate, commonly referred to as soda is a very inexpensive and widely used builder in low cost detergent formulations. Premium detergents use builders like phosphates and/or Zeolites since they have better
25 building properties but are more expensive. There has been continuous work to develop more efficient and faster building systems using less expensive materials. Further, use of phosphates in detergents, is believed by many to be responsible for the eutrophication of rivers and other natural waters bodies. Thus a lot of effort has been made to develop faster building systems using soda as the main raw material.

30 **[0004]** EP0234818 (Unilever, 1987) discloses a detergent composition containing (i) a detergent active system comprising a mixture of (a) an anionic non-soap detergent active; (b) a non-ionic detergent active; and (c) soap; (ii) a watersoluble alkali metal carbonate; and (iii) a water-insoluble particulate carbonate material which is a seed crystal for Calcium carbonate; characterised in a specific combination of weight ratios of the various detergent actives. The builder system in this publication is a mixture of soda and calcium carbonate. There have been many improvements to this technology and many products launched which are improvements over this basic technology where combination of soda
35 and Calcium carbonate is used. The present inventors have determined that the best building systems available presently using sodium carbonate as the basic builder still do not provide the desired fast building and there is scope for improvement on this technology which can be perceived by the consumers in the cleanliness of their washed laundry or in terms of costs of the products.

40 **[0005]** US7186677 (Henkel, 2007) describes a method for producing surfactant granules having good solubility and varying bulk densities comprising (a) providing a mixture of anionic surfactant acids and builder acids having a weight ratio of 1:100 to 1:20 of builder acid to surfactant acid; and (b) contacting the mixture with at least one solid neutralising agent. The builder acid is selected from citric, tartaric, succinic, malonic, adipic, maleic, fumaric, oxalic, gluconic, nitrilotriacetic, aspartic, ethylenediaminetetracetic, among many other acids wherein the builder acid has a particle size below 200 μm .

45 **[0006]** US 3,703,772 discloses a process for drying detergents wherein aqueous solutions of of heat sensitive synthetic organic detergents are dried by admixing such a solution with a finely divided sorbent material, thereby distributing the solution over a greatly increased area, which speeds drying and prevents hot spots during drying and drying the mixed detergent-sorbent-moisture particles.

[0007] US 2,717,243 discloses non caking alkyl sulphonate detergent compositions.

50 **[0008]** CA 979772 discloses detergent compositions for fabric washing.

[0009] Magnesium salt of linear alkyl benzene sulphonic acid is also known and has been used in detergent formulations. US4146551 (Lion, 1979) describes a process for producing the magnesium salt of sulphonic acids and sulfuric esters comprising the step of neutralizing the sulphonic acids and sulphuric esters with an aqueous dispersion containing (1) at least one neutralizing agent selected from the group consisting of magnesium oxide and magnesium hydroxide and (2) at least one neutralizing accelerator selected from the group consisting of benzoic acid, citric acid, malic acid,
55 phosphoric acid, polyphosphoric acid and water soluble salts thereof under a pH of not more than approximately 6. Although this publication discloses use of magnesium based anionic surfactants, it does not teach specific combination of such surfactants with selected builder systems that provides enhanced building in hard water and thereby enhanced

detergency.

[0010] Indian patent IN204326 (Hindustan Lever Ltd, published in 2003) describes a synergistic abrasive cleaning composition containing selective combination of surfactants and a process for producing the same. The process comprises neutralization of at least 40% of the acid precursor of the anionic surfactant using at least one mineral of the dolomites group and mixing abrasives and other conventional ingredients such that the total amount of surfactant is between 0.5 and 35% and the amount of abrasives is 30-95% and processing the mixture in a regular manner. Dolomite is a mineral having the chemical formula $\text{CaMg}(\text{CO}_3)_2$. This prior art publication is directed to hard surface cleaning compositions which comprise Calcium-magnesium salts of anionic surfactants and does not teach combination of magnesium salt of linear alkyl benzene sulphonic acid with selective builder systems for providing enhanced detergency.

[0011] It is an object of the present invention to provide for a solid detergent composition that provides for enhanced cleaning of soiled fabrics as compared to some of the prior art compositions in hard water conditions.

[0012] It is another object of the present invention to provide for a solid detergent composition that provides equal or better cleaning of soiled fabrics at lower cost compared to some of the prior art compositions when cleaned in hard conditions.

SUMMARY OF THE INVENTION

[0013] According to the present invention there is provided a solid detergent composition comprising

- (i) 5 to 90 wt% of magnesium salt of linear alkyl benzene sulphonic acid;
- (ii) 10 to 70 wt% of a water soluble alkali metal carbonate;
- (iii) 3 to 50 wt% of a seed for precipitating calcium carbonate; and
- (iv) an optional co-builder which is a di-carboxylic acid or a salt thereof.

DETAILED DESCRIPTION OF THE INVENTION

[0014] The present inventors have found that use of the specific builder system disclosed in EP0234818 along with a specific co-builder provides for very fast building never before achieved with similar systems.

[0015] Further the present inventors have determined that when a builder system comprising a water soluble alkali metal carbonate and a seed for precipitating calcium carbonate is used along with a specific anionic surfactant viz. magnesium salt of linear alkyl benzene sulphonic acid, the building properties of this builder system combined with the specific properties of this surfactant interact synergistically to provide enhanced cleaning of soiled fabrics especially when washed in hard water.

[0016] The solid detergent composition of the invention comprises magnesium salt of linear alkyl benzene sulphonic acid (Mg-LAS). The solid detergent composition is preferably in the powder, granule, bar or tablet form. The more preferred form of the detergent composition is the powder or granule form. When the detergent composition is present in the powder or granule form, the Mg-LAS therein is preferably present as granules in a particle size range larger than 0.3 mm, more preferably larger than 0.5 mm, most preferably larger than 1 mm, and optimally in the range of 1 to 2 mm. The solid detergent composition comprises Mg-LAS in an amount in the range of 5% to 90%, preferably 10% to 50%, most preferably 15% to 35% by weight of the detergent composition.

[0017] A suitable process to prepare solid form of Mg-LAS is disclosed in our co-pending application 445/MUM/2007 (published as WO2008/107463). The process disclosed and claimed therein comprises the step of neutralization of linear alkyl benzene sulphonic acids with a magnesium based alkali in the presence of 3% to 28% water by weight of the reaction mixture in a high shear mixer. Mg-LAS in very high concentration, as high as 90% in solid forms like powder, granule or extrudable bar can be prepared by the above mentioned process. This process has the advantage that only low amounts of alkali are required for complete neutralisation. Preferred amounts for neutralization are from 5% to 100% stoichiometric excess of the magnesium based alkali. Suitable magnesium based alkali are one or more of magnesium carbonate, magnesium bicarbonate, magnesium oxide, magnesium hydroxycarbonate or magnesium hydroxide.

[0018] A further preferred aspect of the detergent composition of the present invention provides for the Mg-LAS granules to be coated with a water soluble polymer. The water soluble polymers may be poly ethylene oxide, sodium carboxymethyl cellulose, poly vinyl pyrrolidone, poly acrylic acid or poly vinyl alcohol most preferably poly vinyl alcohol. The following processes can be adopted to coat granules of Mg-LAS with water soluble polymers.

[0019] The preferred process for coating of Mg-LAS comprises the steps of spraying a solution of polymer onto Mg-LAS in a pan granulator. The sprayed product may be dried by heating the pan granulator to a high temperature, in the range of 60°C to 90°C to a moisture content of the granules of less than 10 wt%. Alternately, the sprayed product may be transferred to an oven to be dried. A fluidized bed may also be used for coating the polymer with the use of hot air to dry the granules to the desired moisture content. The preferred amount of polymer coating is from 0.25% to 5%, more preferably 0.5% to 2% and most preferably 0.75% to 1.25% by weight of the Mg-LAS granules.

[0020] The solid detergent composition of the invention comprises a water soluble alkali metal carbonate. The alkali metal is preferably sodium or potassium, sodium being preferred. Thus the most preferred alkali metal carbonate is sodium carbonate. The water soluble alkali metal carbonate is present in an amount in the range of 10% to 70%, preferably from 15% to 60%, most preferably from 25% to 50% by weight of the solid detergent composition.

[0021] Another important element of the solid detergent composition of the invention is a seed for precipitating calcium carbonate. By seed for precipitating calcium carbonate is meant a compound which has the ability to act as a seed for precipitation of calcium carbonate in aqueous media. The seed for precipitating calcium carbonate is a substantially water insoluble particulate material. This water insoluble particulate material may be present in the detergent composition or generated *in situ* when the detergent composition is dispersed in water. The more preferred aspect provides for the substantially water insoluble particulate material to be present in the detergent composition. Examples of seed for precipitating calcium carbonate which is generated *in situ* include generation of particulate zinc oxide by including sodium zincate in the detergent composition, generation of particulate alumina by including sodium aluminate or generation of silica by including aluminosilicate in the detergent composition. Suitable substantially water insoluble particulate materials which may be present in the detergent composition are silica, zinc oxide, aluminium oxide, titanium oxide, Zeolite, magnesium oxide or calcium carbonate. Particularly preferred substantially water insoluble particulate material is calcium carbonate. Calcium carbonate may be calcite, or aragonite, most preferably calcite. Calcite is preferably high surface area calcite. Preferably the water insoluble particulate carbonate seed crystal has a surface area greater than 20 m²/g; more preferably greater than 30 m²/g, and most preferably greater than 60 m²/g. The seed for precipitating calcium carbonate is present in an amount in the range of 3% to 50%, more preferably from 5% to 40%, most preferably from 10% to 30% by weight of the solid detergent composition.

[0022] The solid detergent composition optionally and preferably comprises a co-builder which is a di-carboxylic acid or salt thereof. The co-builder present in the solid detergent composition preferably has a water solubility of more than 1 g/l at 25°C. Preferred di-carboxylic acids are oxalic acid, malonic acid and succinic acid, most preferred being oxalic acid. The preferred salts of the di-carboxylic acid are alkali metal or ammonium salts, alkali metal salts being more preferred. The most preferred co-builder is di-sodium oxalate. The co-builder is preferably present in the solid detergent composition as a powder i.e. in a low particle size. The average particle size of the co-builder is preferably less than 150 microns, more preferably less than 75 microns. The co-builder is preferably present in an amount in the range of 1% to 20%, more preferably 5% to 15%, most preferably 5% to 10% by weight of the solid detergent composition.

[0023] The solid detergent composition of the invention preferably comprises an additional builder that is alkali metal silicate. The alkali metal silicate is preferably sodium silicate or potassium silicate, more preferably sodium silicate. Sodium Silicate is a colorless compound of oxides of sodium and silica. It has a range of chemical formulae varying in sodium oxide (Na₂O) and silicon dioxide or silica (SiO₂) contents. It is soluble in water and it is prepared by reacting silica (sand) and sodium carbonate at a high temperature ranging from 1200°C to 1400°C. Aqueous solution of sodium silicate is called water glass. Sodium silicates varying in ratio of Na₂O:SiO₂ from 1:1.6 to 1:4 are known as colloidal silicates. These are usually sold as 20% to 50% aqueous solutions. Of the various types of sodium silicates available the preferred compound to be used in the composition of the invention is neutral sodium silicate. This has a concentration in water in the range of 27% - 39% and a Na₂O:SiO₂ ratio in the range of 3.0 to 3.5. The alkali metal silicate is preferably present in 5% to 50%, more preferably 12% to 40% by weight of the solid detergent composition. The use of the solid detergent composition in 2 g/l to 5 g/l of the wash liquor thereby ensures presence of alkali metal silicate in 0.2 g/l to 1 g/l, preferably 0.5 g/l to 0.8 g/l in the wash liquor.

[0024] According to another aspect of the present invention there is provided a process to prepare a granular detergent composition comprising mixing granules of magnesium salt of linear alkyl benzene sulphonate with powders of a water soluble alkali metal carbonate, seed for precipitating calcium carbonate and a co-builder which is a dicarboxylic acid or salt thereof.

[0025] According to a preferred aspect of the present invention there is provided a process to prepare a detergent composition comprising the steps of:

- (i) preparing granular form of magnesium salt of linear alkyl benzene sulphonate (Mg-LAS) by neutralization of linear alkyl benzene sulphonate with a magnesium based alkali in the presence of 3% to 28% water by weight of the reaction mixture in a high shear mixer;
- (ii) optionally sieving the granules of Mg-LAS to a size range greater than 0.3 mm; and,
- (iii) mixing said granules of Mg-LAS with powder of a water soluble alkali metal carbonate, seed for precipitating calcium carbonate and co-builder, which is a dicarboxylic acid or salt thereof.

[0026] It is preferred that the granules of Mg-LAS used in the above process are coated with a water soluble polymer.

[0027] The invention will now be illustrated with respect to the following non-limiting examples.

ExamplesExamples 1 to 4 and Comparative Example A, B

[0028] These examples illustrate the building kinetics of detergent compositions of the invention compared to conventional products - hand wash protocol.

[0029] Various detergent compositions as shown in Table -1 were prepared and added to hard water of 48 FH (French hardness) using Protocol A as described below. All detergent compositions had a surfactant concentration when in use of 0.7 g/l. The building kinetics was studied by measuring the Ca^{2+} concentration in terms of FH at various time points and the data is presented in Table -2. The method of determining Ca^{2+} concentration is given below:

Measurement of Calcium ion concentration

[0030] The method involved titration with EDTA (di sodium salt of Ethylene Diamine Tetra Acetic acid) using EBT (Eriochrome Black - T) as indicator. About 2 ml of the Calcium ion solution was pipetted out into a 150 ml conical flask. The solution was diluted using 10 ml water. To this was added 5 ml of Ammonia-Ammonium chloride pH 10 buffer. About 35 mg of 1% EBT in potassium nitrate solution was added. A wine red colour was obtained. A standardized EDTA solution was added dropwise from a burette with constant stirring. As more EDTA was added the colour gradually changed from wine red to violet. The end point was identified by a sudden colour change from violet to blue. The calcium ion concentration was calculated using the formula:

[0031] The Ca^{2+} concentration in terms of FH was calculated using the formula:

$$\text{Ca}^{2+} \text{ concentration in FH} = \frac{\text{Ca}^{2+} \text{ ion concentration in ppm}}{4}$$

Protocol A: Protocol used to simulate hand washing.

[0032] The 48 FH water was taken and the detergent composition was added to it and stirred by hand for 45 seconds at about 70-80 rpm. After letting the solution stay for about 5 minutes, the solution was stirred vigorously for about 10 seconds. Samples were then withdrawn at the desired time points using syringes and filtered using a syringe filter into TARSON™ tubes. The filtrate was used to determine Ca^{2+} ion concentration as described earlier.

Table-1

Example	Surfactant	Surfactant, particle size, mm	Soda ash, g/l	HSAC, g/l	STPP, g/l
Comparative Example A	NaLAS	0.5 - 1	1.4	0.3	0.16
Comparative Example B	NaLAS	0.5 - 1	1.5	1.0	-
Example 1	MgLAS	1 - 2	1.5	1.0	-
Example 2	MgLAS	0.5 - 1	1.5	1.0	-
Example 3	MgLAS	1 - 2 PVA coated	1.5	1.0	-
Example 4	MgLAS	0.3 to 0.5	1.5	1.0	-
NaLAS is sodium salt of linear alkyl benzene sulphononic acid HSAC: High Surface Area Calcite with surface area of (20-30) m ² /g. STPP: Sodium tripolyphosphate PVA: Poly Vinyl alcohol					

Table-2

Example	FH, Time = 5 minutes	FH, Time = 10 minutes	FH, Time = 30 minutes
Comparative Example A	24.4	23.3	19.7

(continued)

Example	FH, Time = 5 minutes	FH, Time = 10 minutes	FH, Time = 30 minutes
Comparative Example B	19.1	10.5	8.2
Example 1	0.25	0.15	0.10
Example 2	0.30	0.18	0.15
Example 3	0.28	0.10	0.10
Example 4	4.50	6.80	6.80

[0033] The data in Table - 2 indicates that the detergent compositions of the invention provide for much faster building kinetics as compared to a prior art products.

Examples 5 to 7 and Comparative Examples A, B

[0034] These examples relate to the building kinetics of detergent compositions of the invention compared to conventional samples (Machine wash protocol).

[0035] Compositions of Comparative Example A and B and Example 5 to 7 as shown in Table - 3 below were used to prepare wash liquor that simulates machine wash using a Protocol B as described below:

Protocol B: (Protocol used to simulate washing machine)

[0036] The 48 FH water was taken and the detergent composition was added to it and stirred by using an overhead stirrer at about 160-170 rpm. All detergent compositions had a surfactant concentration when in use of 0.7 g/l. The stirring was continued for a total time of 30 minutes. Samples were withdrawn at the desired time points using syringes and filtered using syringe filter into TARSON™ tubes. The filtrate was used to determine Ca^{2+} concentration as described earlier.

Table-3

Example	Surfactant	Surfactant, particle size, mm	Soda ash, g/l	HSAC, g/l	STPP, g/l	Di-sodium oxalate, g/l
Comparative Example A	NaLAS	0.5 - 1	1.4	0.3	0.16	-
Comparative Example B	NaLAS	0.5 - 1	1.5	1.0	-	-
Example 5	MgLAS	1 - 2	1.5	0.5	-	0.4
Example 6	MgLAS	0.5 - 1.0	1.5	0.5	-	0.4
Example 7	MgLAS	1 - 2; PVA coated	1.5	0.5	-	0.4

[0037] Di sodium oxalate was used in the above experiments in a particle size of < 0.075 mm.

[0038] The data on the building kinetics in terms of FH at various time points using Protocol B for the compositions of Table - 3 is presented in Table - 4 below.

Table-4

Example	FH, Time = 5 minutes	FH, Time = 10 minutes	FH, Time = 30 minutes
Comparative Example A	20.0	21.0	20.0
Comparative Example B	19.7	10.8	9.3
Example 5	4.69	0.10	0.10
Example 6	15.4	14.4	8.10
Example 7	0.30	0.30	0.30

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[0039] The data in Table - 4 indicates that more preferred detergent compositions (Examples 5 and 7) of the invention provide for much faster building kinetics as compared to prior art compositions.

Examples 8 to 12 and Comparative Examples A, B, C

[0040] These relate to the cleaning performance of detergent compositions of the invention compared to conventional samples.

[0041] Compositions of Comparative Examples A to C and Example 8 to 12 as shown in Table - 5 were used to wash various test monitors using a protocol that simulates hand washing. This protocol C is described below.

Protocol C: (Hand wash protocol)

[0042] The 48 FH water was taken and the detergent composition was added to it and stirred by hand at about 50-60 rpm for about 45 seconds. All detergent compositions had a surfactant concentration when in use of 0.7 g/l. The solution was allowed to stand for 5 minutes after which it was stirred by hand for 10 seconds. The fabric test monitors were then added and allowed to soak for 10 minutes. The test monitors were then washed in a tergo-to-meter at 90 rpm for 30 minutes. The test monitors were then rinsed three times at a liquid to cloth ratio of 25 and then dried. The reflectance of the monitors was then measured. Average of reflectance data on three different monitors was taken.

Table-5

Example	Surfactant	Surfactant, particle size, mm	Soda ash, g/l	HSAC, g/l	STPP, g/l
Comparative Example A	NaLAS	0.5 - 1	1.4	0.3	0.16
Comparative Example B	NaLAS	0.5 - 1	1.5	1.0	-
Comparative Example C	NaLAS	0.5 - 1	0.89	0.07	1.02
Example 8	MgLAS	1-2	1.5	1.0	-
Example 9	MgLAS	1-2	1.5	0.7	-
Example 10	MgLAS	0.5 - 1	1.5	0.7	-
Example 11	MgLAS	0.3 - 0.5	1.5	1.0	-
Example 12	MgLAS	0.3 - 0.5	1.5	0.7	-

[0043] The data on the cleaning performance of various test monitors in terms of ΔR^* with respect to the reflectance of the original test monitors is shown in Table - 6. The original (unwashed) test monitors used were WFK10D (composite soil on cotton fabric) had an initial reflectance of about 45, WFK20D (composite soil on poly-cotton fabric) of about 40 and WFK30D (composite soil on polyester fabric) of about 40.

Table-6

Example	ΔR^* WFK10D	ΔR^* WFK20D	ΔR^* WFK30D
Comparative Example A	20.0	18.5	17.6
Comparative Example B	20.0	15.7	17.2
Comparative Example C	22.2	22.9	20.5
Example 8	23.6	28.6	22.7
Example 9	21.3	26.1	21.1
Example 10	20.2	29.2	23.2
Example 11	17.8	22.4	20.3
Example 12	15.2	19.6	22.3

[0044] The data in Table - 6 indicates that the detergent compositions of the invention provide for similar cleaning on some test monitors and better in most other test monitors as compared to the prior art products. The data also indicates

that Mg-LAS of higher particle sizes (Examples 8 to 10) provides for vastly improved cleaning. Further the cleaning of the preferred compositions of the invention (Examples 8 to 10) provide for similar or better cleaning as compared to more expensive Na-LAS+STPP based conventional products.

Examples 13 to 16 and Comparative Examples A and C

[0045] These examples relate to the cleaning performance of more preferred detergent compositions of the invention compared to conventional products.

[0046] Compositions of Comparative Examples A and C and Examples 13 to 16 as shown in Table - 7 were used to wash various test monitors using protocol C as described above.

Table-7

Example	Surfactant	Surfactant, particle size, mm	Soda ash, g/l	HSAC, g/l	STPP, g/l	Oxalic acid, g/l
Comparative Example A	NaLAS	0.5 - 1	1.4	0.3	0.16	-
Comparative Example C	NaLAS	0.5 - 1	0.89	0.07	1.02	-
Example 13	MgLAS	0.3 - 0.12	1.5	0.5	-	0.42
Example 14	MgLAS	1 - 2	1.5	0.5	-	0.42
Example 15	MgLAS	0.5 - 0.3	1.5	0.5	-	0.42
Example 16	MgLAS	1- 2	1.5	0.5	-	0.27

[0047] The particle size of the oxalic acid in the above experiments was in the size range of <0.075 mm. The data on the cleaning performance of various test monitors in terms of ΔR^* with respect to the reflectance of the original test monitors is shown in Table - 8.

Table-8

Example	ΔR^* WFK10D	ΔR^* WFK20D	ΔR^* WFK30D
Comparative Example A	20.0	18.5	17.6
Comparative Example C	22.2	22.9	20.5
Example 13	22.4	22.3	24.2
Example 14	24.5	26.8	25.2
Example 15	24.0	26.8	23.3
Example 16	23.1	27.5	21.6

[0048] The data in Table - 8 indicates that preferred detergent compositions of the invention provide for similar or better cleaning on some test monitors and much better cleaning in most other test monitors as compared to the prior art samples. The data also indicates that Mg-LAS at higher particle sizes > 0.5 mm when combined with co-builder like oxalic acid provides for vastly improved cleaning.

Examples 17 and 18 and Comparative Example C

[0049] These examples relate to cleaning performance of more preferred detergent compositions of the invention as compared to a conventional sample.

[0050] Compositions of Comparative Examples C and Example 17 and 18 as shown in Table - 9 were used to wash various test monitors using a protocol that simulates machine washing. This protocol D is described below:

Protocol D: (Washing machine protocol)

[0051] The 48 FH water was taken in a Tergo-to-meter along with the test monitors and the detergent composition was added to it and stirred for about 10 minutes at 90 rpm. All detergent compositions had a surfactant concentration when in use of 0.7 g/l. The fabric test monitors was then added and allowed to soak for 15 minutes. After this it was run for 30 minutes at 90 rpm. The test monitors were then rinsed three times at a liquid to cloth ratio of 25 and then dried. The reflectance of the monitors was then measured. Average of reflectance data on three different monitors was taken.

[0052] The data on the cleaning performance of various test monitors in terms of ΔR^* with respect to the reflectance of the original test monitors is shown in Table - 10. The original (unwashed) test monitors used were WFK10D (composite soil on cotton fabric) had an initial reflectance of about 45, WFK20D (composite soil on poly-cotton fabric) of about 40 and WFK30D (composite soil on polyester fabric) of about 40.

Table-9

Example	Surfactant	Surfactant particle size, mm	Soda ash, g/l	HSAC, g/l	STPP, g/l	Di-sodium oxalate g/l
Comparative Example C	NaLAS	0.5 - 1	0.89	0.07	1.02	-
Example 18	MgLAS	1 - 2 PVA coated	1.5	0.5	-	0.40
Example 19	MgLAS	1 - 2 PVA Coated	1.5	0.5	-	0.15

[0053] The particle size of the disodium oxalate in the above experiments was in the size range of <0.075 mm.

[0054] The data on the cleaning performance of various test monitors in terms of ΔR^* with respect to the reflectance of the original test monitors is shown in Table - 10.

Table-10

Example	ΔR^* WFK10D	ΔR^* WFK20D	ΔR^* WFK30D
Comparative Example C	26.3	19.4	20.7
Example 17	26.5	26.5	24.2
Example 18	25.1	26.2	25.5

[0055] The data in Table - 10 indicates that preferred detergent compositions of the invention provide for better cleaning on most test monitors as compared to the prior art compositions.

Claims

1. A solid detergent composition comprising:

- (i) 5% to 90% by weight of the detergent composition of magnesium salt of linear alkyl benzene sulphonic acid;
- (ii) 10 to 70% by weight of the detergent composition of a water soluble alkali metal carbonate; and
- (iii) 3 to 50% by weight of the detergent composition of a seed for precipitating calcium carbonate and
- (iv) optionally a co-builder which is a di-carboxylic acid or a salt thereof.

2. A solid detergent composition as claimed in claim 1 wherein water solubility of said co-builder is more than 1 g/l at 25°C.

3. A solid detergent composition as claimed in claim 1 or claim 2 wherein said co-builder has an average particle size less than 150 microns.

4. A solid detergent composition as claimed in any one of the preceding claims wherein said co-builder is present in an amount in the range of 1% to 20 % by weight of the detergent composition.

5. A solid detergent composition as claimed in any one of the preceding claims wherein magnesium salt of linear alkyl

benzene sulphonic acid is present as granules in a particle size range larger than 0.3 mm.

6. A solid detergent composition as claimed in any one of the preceding claims wherein the magnesium salt of linear alkyl benzene sulphonic acid is coated with a water soluble polymer.

7. A solid detergent composition as claimed in any one of the preceding claims wherein said seed for precipitating calcium carbonate is calcium carbonate with a surface area larger than 20 m²/g.

8. A solid detergent composition as claimed in any one of the preceding claims comprising an additional builder that is alkali metal silicate.

9. A process to prepare a solid granular detergent composition comprising mixing granules of magnesium salt of linear alkyl benzene sulphonic acid with powders of a water soluble alkali metal carbonate, a seed for precipitating calcium carbonate and a co-builder which is a dicarboxylic acid or salt thereof.

10. A process as claimed in claim 9 comprising the steps of:

(i) preparing granular form of magnesium salt of linear alkyl benzene sulphonic acid (Mg-LAS) by neutralization of linear alkyl benzene sulphonic acids with a magnesium based alkali in the presence of 3 to 28% water by weight of the reaction mixture in a high shear mixer;

(ii) optionally sieving the granules of Mg-LAS to a size range greater than 0.3 mm; and

(iii) mixing said granules of Mg-LAS with powder of a water soluble alkali metal carbonate, seed for precipitating calcium carbonate and co-builder, which is a dicarboxylic acid or salt thereof.

Patentansprüche

1. Feste Waschmittelzusammensetzung, umfassend:

(i) 5 bis 90 Gewichts-%, bezogen auf die Waschmittelzusammensetzung, Magnesiumsalz von lineares Alkylbenzolsulfonsäure;

(ii) 10 bis 70 Gewichts-%, bezogen auf die Waschmittelzusammensetzung, eines wasserlöslichen Alkalimetallcarbonats und

(iii) 3 bis 50 Gewichts-%, bezogen auf die Waschmittelzusammensetzung, eines Impfkristalls zum Präzipitieren von Calciumcarbonat und

(iv) gegebenenfalls einen Co-Builder, der eine Dicarbonsäure oder ein Salz davon ist.

2. Feste Waschmittelzusammensetzung nach Anspruch 1, wobei die Wasserlöslichkeit des Co-Builders mehr als 1 g/l bei 25 °C ist.

3. Feste Waschmittelzusammensetzung nach Anspruch 1 oder Anspruch 2, wobei der Co-Builder eine durchschnittliche Partikelgröße von weniger als 150 Mikrometer hat.

4. Feste Waschmittelzusammensetzung nach einem der vorangehenden Ansprüche, wobei der Co-Builder in einer Menge im Bereich von 1 bis 20 Gewichts-%, bezogen auf die Waschmittelzusammensetzung, vorliegt.

5. Feste Waschmittelzusammensetzung nach einem der vorangehenden Ansprüche, wobei das Magnesiumsalz von lineares Alkylbenzolsulfonsäure als Körner in einem Partikelgrößenbereich von größer als 0,3 mm vorliegt.

6. Feste Waschmittelzusammensetzung nach einem der vorangehenden Ansprüche, wobei das Magnesiumsalz von lineares Alkylbenzolsulfonsäure mit einem wasserlöslichen Polymer überzogen ist.

7. Feste Waschmittelzusammensetzung nach einem der vorangehenden Ansprüche, wobei der Impfkristall zum Präzipitieren von Calciumcarbonat ein Calciumcarbonat mit einer spezifischen Oberfläche von größer als 20 m²/g ist.

8. Feste Waschmittelzusammensetzung nach einem der vorangehenden Ansprüche, die einen zusätzlichen Builder umfasst, der Alkalimetallsilikat ist.

9. Verfahren zur Herstellung einer festen körnigen Waschmittelzusammensetzung, umfassend Mischen von Körnern von Magnesiumsalz von lineares Alkyl-benzolsulfonsäure mit Pulvern eines wasserlöslichen Alkalimetallcarbonats, einem Impfkristall zum Präzipitieren von Calciumcarbonat und einem Co-Builder, welcher eine Dicarbonsäure oder ein Salz davon ist.

10. Verfahren nach Anspruch 9, umfassend die Schritte:

- (i) Herstellen einer körnigen Form von Magnesiumsalz von lineares Alkyl-benzolsulfonsäure (Mg-LAS) durch Neutralisieren von lineares Alkyl-benzolsulfonsäure mit einem Magnesium-basierten Alkali in Gegenwart von 3 bis 28 Gewichts-% Wasser, bezogen auf das Reaktionsgemisch, in einem Hochschermischer;
- (ii) gegebenenfalls Sieben der Körner von Mg-LAS zu einem Größenbereich, der größer als 0,3 mm ist, und
- (iii) Mischen der Körner von Mg-LAS mit Pulver eines wasserlöslichen Alkalimetallcarbonats, Impfkristall zum Präzipitieren von Calciumcarbonat und Co-Builder, welcher eine Dicarbonsäure oder Salz davon ist.

Revendications

1. Composition détergente solide comprenant :

- (i) 5 % à 90 % en poids de la composition détergente de sel de magnésium d'acide sulfonique d'alkyl benzène linéaire ;
- (ii) 10 à 70 % en poids de la composition détergente d'un carbonate de métal alcalin hydrosoluble ; et
- (iii) 3 à 50 % en poids de la composition détergente d'un germe pour précipiter le carbonate de calcium et
- (iv) en option, un co-adjuvant qui est un acide di-carboxylique ou un sel de celui-ci.

2. Composition détergente solide selon la revendication 1, dans laquelle l'hydrosolubilité dudit co-adjuvant est supérieure à 1 g/l à 25 °C.

3. Composition détergente solide selon la revendication 1 ou la revendication 2, dans laquelle ledit co-adjuvant a une taille de particule moyenne inférieure à 150 microns.

4. Composition détergente solide selon l'une quelconque des revendications précédentes, dans laquelle ledit co-adjuvant est présent dans une quantité dans la plage de 1 % à 20 % en poids de la composition détergente.

5. Composition détergente solide selon l'une quelconque des revendications précédentes, dans laquelle le sel de magnésium d'acide sulfonique d'alkyl benzène linéaire est présent sous forme de granulés dans une plage de tailles de particules supérieure à 0,3 mm.

6. Composition détergente solide selon l'une quelconque des revendications précédentes, dans laquelle le sel de magnésium d'acide sulfonique d'alkyl benzène linéaire est revêtu avec un polymère hydrosoluble.

7. Composition détergente solide selon l'une quelconque des revendications précédentes, dans laquelle ledit germe pour précipiter le carbonate de calcium est du carbonate de calcium ayant une surface supérieure à 20 m²/g.

8. Composition détergente solide selon l'une quelconque des revendications précédentes, comprenant un adjuvant supplémentaire qui est du silicate de métal alcalin.

9. Procédé de préparation d'une composition détergente en granulés solide consistant à mélanger des granulés de sel de magnésium d'acide sulfonique d'alkyl benzène linéaire avec des poudres d'un carbonate de métal alcalin hydrosoluble, un germe pour précipiter le carbonate de calcium et un co-adjuvant qui est un acide dicarboxylique ou sel de celui-ci.

10. Procédé selon la revendication 9, comprenant les étapes consistant à :

- (i) préparer la forme granulaire du sel de magnésium de l'acide sulfonique d'alkyl benzène linéaire (Mg-LAS) grâce à la neutralisation des acides sulfoniques d'alkyl benzène linéaire avec un alcalin à base de magnésium en présence de 3 à 28 % d'eau en poids du mélange réactionnel dans un mélangeur à cisaillement élevé ;
- (ii) tamiser éventuellement les granulés de Mg-LAS au tamis à une plage de tailles supérieures à 0,3 mm ; et

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(iii) mélanger lesdits granulés de Mg-LAS avec la poudre de carbonate de métal alcalin hydrosoluble, le germe pour précipiter le carbonate de calcium et le co-adjuvant, qui est un acide dicarboxylique ou sel de celui-ci.

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REFERENCES CITED IN THE DESCRIPTION

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