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(54) **DETERGENT COMPOSITION**

WASCHMITTELZUSAMMENSETZUNG

COMPOSITION DETERGENTE

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EP-A- 0 110 588 **EP-A- 0 221 777**
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Description

[0001] This invention relates to a detergent composition, in particular to a granular detergent composition containing a non-ionic surfactant and a structuring agent.

[0002] Detergent compositions may be produced by a variety of methods and have historically been produced by a spray-drying process. In view of the recent trend towards detergent powders having a higher bulk density, processes having a mechanical mixing step have been employed to achieve this end. Such powders may be obtained by mixing and densifying a base powder obtained from a spray-drying step in a mixing device and optionally incorporating other components which may be solid or liquid in form. High bulk density powders may also be obtained by mixing and granulating the components of the detergent powder in one or more mixing devices without employing a spray-drying step. In such a densification process, the solid components of the detergent powder are typically bound together by liquid components such as surfactants to form particles which increase in size with continued mixing to form detergent granules. The proportion of liquid to solid has to be controlled otherwise problems such as over-agglomeration may be encountered particularly where high levels of liquid are employed.

[0003] Non-ionic surfactants are a common active component of detergent compositions and are typically incorporated into the composition in liquid form. It is desired to increase the level of active components in detergent powders to improve washing performance. This is however limited by the need to avoid over-agglomeration during processing and to reduce or avoid bleeding during storage of the product. Such problems may occur due to the nonionic surfactant being mobile within the detergent granule and so migrating to the granule surface.

[0004] Following mechanical mixing to form detergent granules, the granules may then be dried for example in a fluid bed if desired. This is typically carried out at elevated temperature. However, at high temperatures a liquid component of the detergent granule for example non-ionic surfactant may become more flowable and so bleed out from within the granule. The granule thus becomes sticky and prone to over-agglomeration which may cause the drying apparatus to foul. Furthermore, an undesirable loss of active component from the granule may be observed. The maximum temperature at which the drying apparatus may be operated is limited by the wish to reduce such effects. Bleeding of non-ionic surfactant from the granule after packaging also presents a significant drawback in that unsightly marks may appear on the packaging and caking may occur.

[0005] The problem of over agglomeration has previously been addressed by incorporating a viscosity raising component into the composition. EP-A-544 365 (Unilever) discloses that agglomeration control may be achieved by increasing the viscosity of the total liquid composition and discloses the use of water and soap formed in situ to achieve this. EP-A-544 492 discloses that powder flow properties may be improved by including soap in a detergent composition as a structurant.

[0006] EP 622 454, filed on 30 April 1993 and published on 2 November 1994, discloses a process for making a granular laundry detergent composition or component by dissolving a structuring agent in a nonionic surfactant and dispensing the resultant mixture on a powder. The mixture has a viscosity of at least 350 mPas at a shear rate of 25 sec⁻¹ at the operating temperature. Polyvinyl alcohols, polyhydroxyacrylic acid polymers, polyvinyl pyrrolidone, PVNO, sugars, artificial sweeteners and their derivatives are disclosed as being suitable for use. Polyvinyl pyrrolidine and polyvinyl pyridine N oxide are exemplified as structuring agents and sprayed onto a powder at a temperature of between 15 and 40°C.

[0007] EP 622 454 is concerned with the problem of improving the storage stability and physical properties of granular detergents which are rich in non-ionic surfactant. The problems of reducing fouling and facilitating a higher drying temperature in the production process are not addressed.

[0008] We have surprisingly found that a detergent composition containing a non-ionic surfactant and an organic polymeric structuring agent having certain viscosity characteristics may ameliorate problems due to over-agglomeration and fouling of drying apparatus and also present other unexpected advantages.

[0009] A first aspect of the invention provides a granular detergent composition comprising (i) a nonionic surfactant, preferably in an amount of at least 10% by weight, (ii) a detergency builder, and (iii) a water-soluble polyvinyl alcohol in an amount of no more than 5% by weight of the total detergent composition, in which a mixture of (a) 1 part of a 10% by weight aqueous solution of the polyvinyl alcohol, and (b) 2 parts of a nonionic surfactant mixture comprising a 1:1 ratio of a C₁₃₋₁₅ alcohol ethoxylate having an average of 3 ethoxylate units and a C₁₃₋₁₅ alcohol ethoxylate having an average of 6.5 ethoxylate units, has a viscosity of at least 1.5 Pa.s at 40°C and a shear rate of 50 sec⁻¹.

[0010] A second aspect of the present invention provides for the use of a polyvinyl alcohol as characterised in the first aspect in a granular detergent composition or component to reduce or avoid fouling of apparatus for producing the said composition or component at, especially elevated temperature. Suitably, the polyvinyl alcohol also serves to reduce or prevent the mobility of nonionic surfactant in the composition, especially at elevated temperatures.

[0011] The invention also provides a process for the production of a granular detergent composition or component according to the first aspect in which granules of the composition or component are subjected in a drying zone to a temperature in excess of 45°C which process comprises incorporating the water-soluble polyvinyl alcohol into the

composition or component comprising a nonionic surfactant and a detergency builder such that fouling of the drying zone is reduced or avoided.

[0012] The composition or component is suitably subjected to a temperature of at least 50°C and desirably at least 60°C in the drying zone to provide efficient removal of water. The drying zone, for example a fluid bed dryer, is suitably

fed with an air stream at an air temperature of at least 70°C, preferably at least 80°C, and more preferably at least 90°C.

[0013] All viscosities quoted herein are measured at 40°C and a shear rate of 50 sec⁻¹ unless otherwise stated. All

% by weight values quoted herein are based on the total detergent composition unless otherwise stated.

[0014] Preferably, the viscosity of the nonionic surfactant and aqueous polymer solution mixture is at least 2 Pa.s and more preferably at least 2.3 Pa.s. It is further preferred that the said mixture has a viscosity of at least 0.8 and more preferably at least 1.0 Pa.s at 40°C and a shear rate of 390 sec⁻¹.

[0015] The viscosity characteristics of the structuring agent in combination with the nonionic surfactant provides granules of a smaller particle size as compared to similar granules without the structuring agent. A higher level of liquid, for example nonionic surfactant, may be incorporated into a granule of a given size. Further, the point at which granulation is terminated during the production process is readily controllable and over-agglomeration may be reduced or avoided.

[0016] It is preferred that the structuring agent has a relatively high viscosity at higher temperatures as well as at 40°C. In particular the structurant is such that the nonionic/structurant mixture referred to in the first aspect of the invention preferably has a viscosity of at least 0.8 and especially at least 1.0 Pa.s at 60°C and a shear rate of 50 sec⁻¹. High viscosity also permits a greater level of nonionic surfactant to be incorporated into the granule which has the practical benefit of providing an increased detergency effect in the wash without there being any significant drawback in processing the composition. Moreover, the inclusion of higher levels of nonionic surfactant is facilitated by the reduced tendency to fouling during processing or bleeding when packaged.

[0017] The structuring agent is polyvinyl alcohol. It is preferred that the structuring agent has hydrogen bonding capability. Without wishing to be bound by any theory, it is believed that the intermolecular interactions between the structuring agent and the nonionic surfactant facilitate the retention of the surfactant within the granule and thus immobilising the nonionic surfactant and reducing bleeding.

[0018] In order to obtain a combination of properties a mixture of polymers may be employed for example polyvinyl alcohol and CP5 ex BASF. The polymer suitably has a molecular weight of at least 20000, preferably at least 50000 and especially at least 100000.

[0019] PVA provides a desirable combination of advantages. Desirably the PVA has a molecular weight of at least 100000, and preferably at least 120000. Accordingly a further aspect of the invention provides for the use of polyvinyl alcohol as a structurant in a granular detergent composition.

[0020] Where a drying apparatus, for example a fluid bed is employed in the production process, the detergent composition may be subjected to elevated temperatures for example in excess of 50°C in order to remove moisture from the composition. At such temperatures components of the composition may soften, melt or increase in mobility within the granule and cause processing difficulties.

[0021] A further aspect of the invention provides a granular detergent composition comprising a nonionic surfactant, a detergency builder and a structuring agent comprising an organic polymeric material having a melting point of at least 70°C, preferably at least 85°C and more preferably at least 100°C.

[0022] The structuring agent employed in this aspect of the invention suitably has the same viscosity characteristics as a structuring agent suitable for use in a composition according to the first aspect of the invention.

[0023] By employing a structuring agent which has a melting point above the operating temperature in the drying apparatus, the agent remains in the solid phase and the structuring effect is retained thus reducing processing difficulties.

[0024] It is preferred that the structuring agent is relatively viscous at higher temperatures as well as at 40°C. Such viscosity characteristics are preferred as at higher temperatures bleeding of liquid components, particularly nonionic surfactant, from within the granule may occur as the liquid components have greater mobility thus loss of active and over-agglomeration may be reduced.

[0025] A further practical advantage of employing such a structuring agent is that the drying apparatus may be operated at a higher temperature whereby water is driven out of the detergent granules thus providing a detergent composition having a low relative humidity.

[0026] Yet another aspect of the invention provides a granular detergent composition comprising a nonionic surfactant, a detergency builder and a structuring agent comprising an organic polymeric material having a melting point of at least 70°C wherein the composition has a relative humidity not in excess of 30%, preferably not in excess of 20% and more preferably not in excess of 10%.

[0027] Relative humidity is a measurement of the water activity in a solid material. It is the ratio of the current water concentration in the air (kg water/kg air) to the maximum at a given temperature and pressure expressed as a percentage of the value of saturated air. All relative humidity values quoted herein are under conditions of 1 atmosphere

and 37°C using a Novasina relative humidity meter unless otherwise stated.

[0028] Low relative humidity composition according to the invention permit moisture sensitive components to be admixed therewith and retain an acceptable level of storage stability.

[0029] Preferably the granules of composition according to the invention have an average Rosin-Rammler (RR) diameter not in excess of 1000 µm and more preferably not in excess of 900 µm.

[0030] Where a drying step, for example in a fluid bed is employed in the production, it is preferred that the RR diameter before and after drying increases by less than 100 µm, more preferably by less than 50 µm and desirably the RR diameter decreases.

[0031] The lower particle size is preferred as greater agglomeration control is possible.

[0032] Suitably granules of a composition according to the invention have a high Youngs Modulus, preferably at least 5 MPa and especially at least 10 MPa. Granules according to the present invention having a high Youngs Modulus possess excellent dynamic flow rate properties. It is believed that the improved dynamic flow rate is due to the viscosity characteristics of the structuring agent which provide particles having increased particle strength.

[0033] Suitably, the structuring agent is incorporated into the composition as a pumpable aqueous solution or dispersion of an organic polymeric material preferably by premixing the solution or dispersion with other liquid components of the composition for example non-ionic surfactant and then mixing with the solid components. If desired the structuring agent may be incorporated as a solid.

[0034] The aqueous solution or dispersion may comprise from 1 to 50%, preferably 5 to 40% and especially 5 to 30% by weight of polymer. Introduction of the polymer in solution or dispersion facilitates mixing of the polymer with other components of the composition.

[0035] Nonionic surfactants that may be employed in the composition include the primary and secondary alcohol ethoxylates, especially the C₈-C₂₀ aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C₁₀-C₁₅ primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglycosides also glycerol monoethers, and polyhydroxyamides (glucamide).

[0036] Suitably the weight ratio of nonionic surfactant to the structuring agent in the final composition is 1:1 to 50:1, preferably 3:1 to 30:1, and more preferably 5:1 to 25:1.

[0037] We have found that an excellent structuring effect may be secured and problems of bleeding and processing difficulties may be reduced by incorporating an electrolyte into the detergent compositions according to the present invention.

[0038] Accordingly a further aspect of the invention provides a detergent composition according to any aspect of the present invention which further comprises an electrolyte.

[0039] The invention also provides for the use of a combination of an organic polymeric material and an electrolyte in a detergent composition as a structuring agent. Such a combination may be used advantageously to reduce or avoid fouling in the apparatus for producing the said composition.

[0040] Suitably the electrolyte comprises an alkali metal salt preferably a sodium salt of carbonate, silicate, sulphate and/or citrate. Salts of carbonate and/or citrate are especially preferred as they provide enhanced benefits as regards reducing or avoiding fouling of drying apparatus at high temperature in addition to their known benefits. Without wishing to be bound by any theory, it is believed that the contribution of salt, especially citrate and carbonate, and the polymeric material act to reduce the number of pores within the granule and so reduce the mobility of the mobile components within the granule.

[0041] The salt, especially carbonate and/or citrate may suitably be present in an amount of 1 to 60 wt%, preferably from 2 to 40 wt% and especially 5 to 20%.

[0042] It is highly desirable that the compositions according to the invention comprises, PVA, citrate and Sokolan HP22.

[0043] Compositions according to the present invention may optionally contain bleaching components and other active ingredients to enhance performance and properties.

[0044] The surfactants may be chosen from soap and non-soap anionic, cationic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof. Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch. It is preferred however that soap is not present in the composition as the soap now acts as a structurant and we prefer that the structuring effect be due substantially wholly to the organic polymer.

[0045] Anionic surfactants are well-known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl chain length of C₈-C₁₅; primary and secondary alkyl sulphates, particularly C₁₂-C₁₅ primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

[0046] The choice of detergent-active compound (surfactant), and the amount present, will depend on the intended use of the detergent composition. For example, for machine dishwashing a relatively low level of a low-foaming nonionic

surfactant is generally preferred. In fabric washing compositions, different surfactant systems may be chosen, as is well known to the skilled formulator, for handwashing products and for products intended for use in different types of washing machine.

[0047] The total amount of surfactant present will also depend on the intended end use and may be as low as 0.5 wt%, for example, in a machine dishwashing composition, or as high as 60 wt%, for example, in a composition for washing fabrics by hand. In compositions for machine washing of fabrics, an amount of from 5 to 40 wt% is generally appropriate.

[0048] The composition will generally also contain one or more detergency builders. The total amount of detergency builder in the compositions will suitably range from 10 to 80 wt%, preferably from 15 to 60 wt%.

[0049] Inorganic builders that may be present include sodium carbonate, if desired in combination with a crystallisation seed for calcium carbonate, as disclosed in GB 1 437 950 (Unilever); crystalline and amorphous aluminosilicates, for example, zeolites as disclosed in GB 1 473 201 (Henkel), amorphous aluminosilicates as disclosed in GB 1 473 202 (Henkel) and mixed crystalline/amorphous aluminosilicates as disclosed in GB 1 470 250 (Procter & Gamble); and layered silicates as disclosed in EP 164 514B (Hoechst). Inorganic phosphate builders, for example, sodium orthophosphate, pyrophosphate and tripolyphosphate, may also be present, but on environmental grounds those are no longer preferred.

[0050] Zeolite builders may suitably be present in amounts of from 10 to 70 wt%, amounts of from 15 to 50 wt% being especially suitable for (machine) fabric washing compositions. The zeolite used in most commercial particulate detergent compositions is zeolite A. Advantageously, however, maximum aluminium zeolite P (zeolite MAP) described and claimed in EP 384 070A (Unilever) may be used. Zeolite MAP is an alkali metal aluminosilicate of the P type having a silicon to aluminium ratio not exceeding 1.33, preferably not exceeding 1.15, and more preferably not exceeding 1.07.

[0051] Organic builders that may be present include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphinates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, alkyl- and alkenylmalonates and succinates; and sulphonated fatty acid salts. This list is not intended to be exhaustive.

[0052] Especially preferred organic builders are citrates, nitrolotriacetic acid and oxydisuccinate and are suitably used in amounts of from 5 to 30 wt%, preferably from 10 to 25 wt%; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, preferably from 1 to 10 wt%.

[0053] Builders, both inorganic and organic, are preferably present in alkali metal salt, especially sodium salt, form.

[0054] The detergent composition is suitably produced by a spray-dry method having a subsequent mixing step in which the polymer is incorporated or preferably a non tower method. The non tower method is particularly useful where a detergent composition having a high bulk density is required. The crude composition may be produced by a non tower granulation process, for example as described in EP 340 013, EP 367339, EP 390351 and EP 420317 (Unilever).

[0055] A further aspect of the invention provides a process for the production of a composition according to the invention comprising mixing an aqueous solution or dispersion of a polymeric material with a nonionic surfactant mixing the mixture with a solid builder to form a particulate detergent composition wherein the nonionic/polymer mixture has a viscosity of at least 1.5 Pa.s at 40°C and a shear rate of 50 sec⁻¹.

[0056] Where a continuous granulation process is employed to produce the crude composition mixing and densification steps may be carried out simultaneously using a high speed mixer, suitable examples include a Shugi (trademark) Granulator, a Drais (trade mark) K-TTP 80 Granulator and the Lodige (trade mark) CB30 recycler. The residence time in the mixing step is suitably about 5 to 30 seconds and the rate of mixing in the apparatus is suitably in the range 100 to 2500rpm depending upon the degree of densification and the particle size required. A granulation step may be employed if desired and may be carried out using a lower speed mixer for example, the Drais (trade mark) K-T 160 and the Lodige (trade mark) KM300 mixer. The residence time in the granulation step is suitably about 1 to 10 minutes and the rate of mixing in the apparatus is about 40 to 160 rpm.

[0057] Detergent compositions produced by the process of the invention are preferably admixed with a bleach system. Machine dishwashing compositions may suitably contain a chlorine bleach system, while fabric washing compositions may more desirably contain peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, capable of yielding hydrogen peroxide in aqueous solution.

[0058] Suitable peroxy bleach compounds include organic peroxides such as urea peroxide, and inorganic persalts such as the alkali metal perborates, percarbonates, perphosphates, persilicates and persulphates. Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate.

[0059] The peroxy bleach compound is suitably present in an amount of from 5 to 35 wt%, preferably from 10 to 25 wt%.

[0060] The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. The bleach precursor is suitably present in an amount of from 1 to 8 wt%, preferably from 2 to 5 wt%.

[0061] Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and peroxybenzoic acid precursors; and peroxycarbonic acid precursors. An especially preferred bleach precursor suitable for use in the present invention is N,N,N',N'-tetracetyl ethylenediamine (TAED).

[0062] The novel quaternary ammonium and phosphonium bleach precursors disclosed in US 4 751 015 and US 4 818 426 (Lever Brothers Company) and EP 402 971A (Unilever) are also of great interest. Especially preferred are peroxycarbonic acid precursors, in particular cholyl-4-sulphophenyl carbonate. Also of interest are peroxybenzoic acid precursors, in particular, N,N,N-trimethylammonium toluoyloxy benzene sulphonate; and the cationic bleach precursors disclosed in EP 284 292A and EP 303 520A (Kao).

[0063] A bleach stabiliser (heavy metal sequestrant) may also be present. Suitable bleach stabilisers include ethylenediamine tetraacetate (EDTA) and the polyphosphonates such as Dequest (Trade Mark), EDTMP.

[0064] An especially preferred bleach system comprises a peroxy bleach compound (preferably sodium percarbonate optionally together with a bleach activator), and a transition metal bleach catalyst as described and claimed in EP 458 397A, EP 458 398A and EP 509 787A (Unilever).

[0065] Other materials that may be present include sodium silicate and sodium metasilicate; antiredeposition agents such as cellulosic polymers; fluorescers; inorganic salts such as sodium sulphate; lather control agents or lather boosters as appropriate; proteolytic and lipolytic enzymes; dyes; coloured speckles; perfumes; foam controllers; and fabric softening compounds. This list is not intended to be exhaustive.

[0066] The invention is illustrated by the following nonlimiting Examples.

Example 1

[0067] Aqueous polymer solutions were prepared and mixed with a 1:1 mixture of i) nonionic active having 3 ethoxylate groups and ii) nonionic active having 6.5 ethoxylate groups such that the mixture had a liquid moisture content of 35% that is approximately 1 part polymer solution to 2 parts nonionic mixture. The viscosity of the mixture was then measured using a Haake RV20 by heating the liquid to 40°C, centrifuging to extract air, reheated to 40°C and measured at a shear rate of 50sec⁻¹ and 390 sec⁻¹. As a reference a similar solution using water instead of the polymer solution was also prepared and measured. The results are recorded in Table 1.

Table 1

	Viscosity (Pa.s) Shear 50sec ⁻¹	Viscosity(Pa.s) Shear390sec ⁻¹
Polyvinyl alcohol (10% in water)	2.5	0.8
Polyvinyl pyrrolidone (10% in water comparative)	3.1	0.9
dextrin (20% in water comparative)	2.9	1.2
water (comparative)	1.2	0.7
PVA :MOWIOL 18-88 ex Hoechst PVP :PVP-40 (Mwt 40000) ex Sigma Dextrin weiss ex Merck		

[0068] The higher viscosity of the polymer/nonionic solutions provides improved structuring which improves powder flow properties, permits higher process temperatures without fouling and facilitates the inclusion of higher levels of nonionic surfactant in compositions of the invention.

Examples 2 to 4 and Comparative Example A

[0069] Compositions were prepared by mixing the components listed in Table 2 in a Lodige CB30 "Recycler" mixer. The liquid phase components were premixed and sprayed onto the other components. The mixture was then layered using a zeolite to prevent further granulation and subjected to further mixing in a Lodige "Ploughshare" mixer at 120rpm with the "choppers" on. In some cases the granules were passed through a fluid bed having a back mix section, and first and second plug flow sections for drying and cooling respectively. The air flow in each section was 950, 1000, and 950 kg/hr respectively.

Table 2

Example	2	3	4	A
Sodium carbonate	151	151	147	148
Zeolite 4A	499	497	498	499
PAS adjunct	212	212	216	216
NI 3EO	105	100	74	72
NI 6.5EO	0	0	83	86
NI 7EO	81	81	0	0
Fatty acid/ NI3EO (1:1)	0	0	0	54
NaOH (50%)	0	0	0	25
Water	0	0	0	96
PVA (10%)	90	90	112	0
CB30(rpm)	1600	1800	1500	1500
Zeolite 4A (Layering)	32	32	30	30
KM300weir (% open)	80	80	60	60
Back mix (°C)	100	110	-	-
Drying (°C)	90	120	-	-
Cooling(°C)	16	16	-	-
all figures are flows (kg/hr) PAS adjunct is a particle comprising PVA - polyvinyl alcohol MOWIOL 18-88 ex Hoechst				

[0070] The compositions obtained in Example 4 and Comparative A ex CB30 and KM300 were inspected to assess the effect of PVA as compared to soap (produced from the fatty acid/ sodium hydroxide mixture). Example 4 showed a fine non-sticky powder from both mixers whereas Comparative A produced an unstable powder ex CB30 and large sticky particles ex KM 300. This illustrates that compositions structured with PVA provide powders which do not over agglomerate and which possess good powder properties as compared to a soap structured powder.

Example 5

[0071] Various parameters of Example 2 and 3 compositions were tested and are recorded in Table 3.

Table 3

Example	2	3
Bulk density ex CB30 (g/l)	699	681
BD ex KM 300 (g/l)	779	753
BD ex fluid bed (g/l)	836	814
Rosin Rammler diameter ex KM 300 (μm)	649	500
Rosin Rammler diameter ex fluid bed (μm)	686	549
Dynamic flow rate	115	129

[0072] Compositions according to the invention have good powder flow properties and the relatively small increase in diameter due to the fluid bed is beneficial in avoiding fouling. Visual inspection of the mixers and fluid bed showed no signs of fouling at both 90°C (Example 2) and 120° (Example 3).

Examples 6 and 7 and Comparative Examples B and C

[0073] Compositions having components as listed in Table 4 were produced by mixing the premixed liquid components with the solid components in a Lodge CB 30 "Recycler" mixer at 1200 rpm to obtain a granular material. For Examples 6 and Comparative B the material was then dried at 85°C for 30 minutes in a batch fluid bed, allowed to cool and properties measured. Examples 7 and C were fed to a Lodge KM 300 "Ploughshare" mixer (weir 80% open) from the "Recycler" prior to drying under the same regime as Examples 6 and B.

Table 4

Example	6	B	7	C
Zeolite 4A	619	636	650	650
Sodium carbonate	178	184	188	188
Nonionic 7EO	189	176	171	170
Nonionic 3EO	63	58	55	53
Free water	0	126	0	125
Aqueous PVA solution (10%)	151	0	137	0
DFR (ml/s) (before drying)	57	62	-	-
DFR (ml/s) (after drying)	98	79	-	-
RRdiameter μm (before drying)	789	1041	718	847
RRdiameter μm (after drying)	875	1413	668	906

[0074] all figures are flow (kg/hr)

[0075] The results in Table 4 illustrate that a PVA structured composition gives smaller particles than a water structured composition. This is due to the higher viscosity of the NI/PVA than of NI/water and provides a beneficial increase in the dynamic flow rate of the composition.

Example 8 to 11 and Comparative D

[0076] Composition 8 to 11 and composition D, as a control, were prepared having the components listed in Table 5 with Examples 8 to 11 having a different polymer and D not having the polymer component by dry mixing the solid materials in a Moulinette 094 260 W mixer for 3 seconds, adding the liquids (20% aqueous solution of polymer) and mixing for 7 seconds and then granulating for 4 seconds. The compositions were then dried in an Aeromatic Strea-1 fluid bed at 85°C for 45 minutes with an air flow of 40m³/hr. The Young's Modulus of the compositions were measured by compressing the composition into a tablet in a RIIC 25 ton ring press until liquid leakage first occurred. The Young's Modulus was measured using an Instron 1140 at a compression speed of 1mm/min and results are recorded in Table 6.

Table 5

	Parts by weight
Zeolite (wessalith P)	53.05
Sodium carbonate	15.30
Nonionic 6.5EO (Imbetin)	17.58
Nonionic 3EO (Lorodac)	5.86
Water	12.62
Polymer	3.16 (0 for composition D)

Table 6

Example	Polymer	Young's Modulus (MPa)
8	PVA	16

Table 6 (continued)

Example	Polymer	Young's Modulus (MPa)
9 (comparative)	Polyvinyl pyrrolidone	5
10 (comparative)	dextrin	20
11 (comparative)	starch	10
D (comparative)	water	<5

[0077] Compositions 8 to 11 which are structured with organic polymers have a higher Young's Modulus than an equivalent composition D which is structured with water and they possess improved flow properties.

Examples 12 to 14 and Comparative Example E

[0078] A series of compositions were produced by the process described in Examples 2 and 3. The compositions contained the following components (by weight):

Table 7

Example	12	13	14	E
*PAS adjunct	23	21	20	21
Zeolite A24	52	48	47	47
Nonionic 7EO	24	21	21	21
Tri Sodium Citrate	8	6	6	6
Polymer solution/dispersion (10% polymer aqueous solution)	10	10	10	10 (water)

* contains approximately 45% primary alkyl sulphate, 50% zeolite A24 and 5% sodium carbonate.

[0079] The air and powder temperature in the fluid bed was increased for each Example to the maximum attainable without causing fouling. The powder temperature was also measured. The results are shown in Table 8.

Table 8

Example	Polymer	T _{air} (°C)	T _{power} (°C)
12	PVA	100	64
13 (comparative)	PVP-40	70	50
14 (comparative)	CP5	80	52
E (comparative)	water	70	45

[0080] By employing a polymer as a structuring agent, higher drying temperatures may be achieved without fouling of the fluid bed. PVA is especially effective in facilitating higher drying temperatures. With an air temperature of 100°C excellent water removal from Example 12 was observed providing a powder having a low moisture content.

Claims

1. A granular detergent composition or component comprising (i) a nonionic surfactant, (ii) a detergency builder, and (iii) a water-soluble polyvinyl alcohol, the water-soluble polyvinyl alcohol amounting to no more than 5% by weight of the total detergent composition, in which a mixture of

- 1 part of a 10% by weight of an aqueous solution of the polyvinyl alcohol, and
- 2 parts of a nonionic surfactant mixture comprising a 1:1 ratio of a C₁₃₋₁₅ alcohol ethoxylate having an average of 3 ethoxylate units and a C₁₃₋₁₅ alcohol ethoxylate having an average of 6.5 ethoxylate units, has a viscosity of at least 1.5 Pa.s at 40°C and a shear rate of 50 s⁻¹, and at least 0.8 Pa.s at 60°C and a shear rate of 50 s⁻¹.

2. A composition or component according to claim 1 in which the mixture of (a) and (b) has a viscosity of at least 0.8 Pa.s at 40°C and a shear rate of 390 s⁻¹.
3. A composition or component according to claim 1 or 2 in which the polyvinyl alcohol has a molecular weight of at least 50000.
4. A composition or component according to claim 3 in which the polyvinyl alcohol has a molecular weight of at least 100000.
5. A composition or component according to any preceding claim which further comprises an electrolyte selected from a citrate and/or carbonate salt.
6. A composition or component according to any preceding claim in which the polyvinyl alcohol has a melting point of at least 70°C.
7. Use of a water-soluble polyvinyl alcohol in a granular detergent composition or component comprising a nonionic surfactant and a detergency builder to reduce or avoid fouling of apparatus for producing the said composition or component, wherein the polyvinyl alcohol amounts to no more than 5% by weight of the total detergent composition, and wherein the polyvinyl alcohol is such that a mixture of
 - (a) 1 part of a 10% by weight of an aqueous solution of the polyvinyl alcohol, and
 - (b) 2 parts of a nonionic surfactant mixture comprising a 1:1 ratio of a C₁₃₋₁₅ alcohol ethoxylate having an average of 3 ethoxylate units and a C₁₃₋₁₅ alcohol ethoxylate having an average of 6.5 ethoxylate units, has a viscosity of at least 1.5 Pa.s at 40°C and a shear rate of 50 s⁻¹, and of at least 0.8 Pa.s at 60°C and a shear rate of 50 s⁻¹.
8. Use of a polyvinyl alcohol as defined in claim 7 to reduce or avoid fouling in a fluid bed dryer at elevated temperature.
9. A process for the production of a granular detergent composition or component according to claim 1 in which granules of the composition or component are subjected in a drying zone to a temperature in excess of 45°C which process comprises incorporating the water-soluble polyvinyl alcohol into the composition or component comprising a nonionic surfactant and a detergency builder such that fouling of the drying zone is reduced or avoided.

Patentansprüche

1. Granuläre Waschmittelzusammensetzung oder -Komponente, umfassend (i) ein nichtionisches Tensid, (ii) einen Waschmittelbuilder und (iii) einen wasserlöslichen Polyvinylalkohol, wobei der wasserlösliche Polyvinylalkohol sich auf eine Menge von nicht mehr als 5 Gewichtsprozent auf die gesamte Waschmittelzusammensetzung beläuft, wobei ein Gemisch von
 - (a) 1 Teil einer 10-gewichtsprozentigen wässrigen Lösung des Polyvinylalkohols und
 - (b) 2 Teilen eines nichtionischen Tensidgemisches, umfassend ein 1:1-Verhältnis von einem C₁₃₋₁₅-Alkoholethoxylat mit durchschnittlich 3 Ethoxylateinheiten und einem C₁₃₋₁₅-Alkoholethoxylat mit durchschnittlich 6,5 Ethoxylateinheiten, eine Viskosität von mindestens 1,5 Pa.s bei 40°C und einer Schergeschwindigkeit von 50 s⁻¹ und mindestens 0,8 Pa.s bei 60°C und einer Schergeschwindigkeit von 50 s⁻¹ aufweist.
2. Zusammensetzung oder Komponente nach Anspruch 1, wobei das Gemisch von (a) und (b) eine Viskosität von mindestens 0,8 Pa.s bei 40°C und einer Schergeschwindigkeit von 390 s⁻¹ aufweist.
3. Zusammensetzung oder Komponente nach Anspruch 1 oder 2, wobei der Polyvinylalkohol ein Molekulargewicht von mindestens 50000 aufweist.
4. Zusammensetzung oder Komponente nach Anspruch 3, wobei der Polyvinylalkohol ein Molekulargewicht von mindestens 100000 aufweist.
5. Zusammensetzung oder Komponente nach einem vorangehenden Anspruch, die weiterhin einen Elektrolyten,

ausgewählt aus einem Citrat- und/oder Carbonatsalz, umfasst.

6. Zusammensetzung oder Komponente nach einem vorangehenden Anspruch, wobei der Polyvinylalkohol einen Schmelzpunkt von mindestens 70°C aufweist.

7. Verwendung eines wasserlöslichen Polyvinylalkohols in einer granulären Waschmittelzusammensetzung oder -Komponente, umfassend ein nichtionisches Tensid und einen Waschmittelbuilder, um Ablagerungen in der Anlage zur Herstellung der Zusammensetzung oder Komponente zu vermindern oder zu vermeiden, wobei sich der Polyvinylalkohol auf nicht mehr als 5 Gewichtsprozent der gesamten Waschmittelzusammensetzung beläuft und wobei der Polyvinylalkohol so ausgelegt ist, dass ein Gemisch von

- (a) 1 Teil einer 10-gewichtsprozentigen wässrigen Lösung des Polyvinylalkohols und
(b) 2 Teilen eines nichtionischen Tensidgemisches, umfassend ein 1:1-Verhältnis von einem C₁₃₋₁₅-Alkoholethoxylat mit durchschnittlich 3 Ethoxylateinheiten und einem C₁₃₋₁₅-Alkoholethoxylat mit durchschnittlich 6,5 Ethoxylateinheiten,
eine Viskosität von mindestens 1,5 Pa.s bei 40°C und einer Schergeschwindigkeit von 50 s⁻¹ und von mindestens 0,8 Pa.s bei 60°C und einer Schergeschwindigkeit von 50 s⁻¹ aufweist.

8. Verwendung eines wie in Anspruch 7 definierten Polyvinylalkohols, um Ablagerungen in einem Wirbelschichttrockner bei erhöhter Temperatur zu vermindern oder zu vermeiden.

9. Verfahren zur Herstellung einer granulären Waschmittelzusammensetzung oder -komponente nach Anspruch 1, wobei die Granulen der Zusammensetzung oder Komponente in einer Trockenzone einer Temperatur oberhalb 45°C unterzogen werden, wobei das Verfahren Einarbeiten des wasserlöslichen Polyvinylalkohols in die Zusammensetzung oder Komponente, umfassend ein nichtionisches Tensid und einen Waschmittelbuilder, derart umfasst, dass die Bildung von Ablagerungen in der Trockenzone vermindert oder vermieden wird.

Revendications

1. Composition ou composant détergent granulaire comprenant (i) un tensioactif non ionique, de préférence dans une quantité d'au moins 10 % en masse, (ii) un édificateur de détergence, et (iii) un alcool polyvinylique soluble dans l'eau dans une quantité ne dépassant pas 5 % en masse de la composition détergente totale, dans laquelle le mélange de

- (a) 1 partie de solution aqueuse à 10 % en masse de l'alcool polyvinylique et de
(b) 2 partie du mélange d'agent tensioactif comprenant une partie d'éthoxylat d'alcool en C13 - C15 ayant en moyenne 3 unités d'éthoxylat, et d'éthoxylat d'alcool en C13 - C15 ayant en moyenne 6,5 unités d'éthoxylat,

ayant une viscosité d'au moins 1,5 mPas à 40°C et un taux de cisaillement de 50 sec⁻¹.

2. Composition ou composant selon la revendication 1, dans lequel le mélange de (a) et de (b) a une viscosité d'au moins 0,8 Pa.s à 40°C et un taux de cisaillement de 390 s⁻¹.

3. Composition ou composant selon la revendication 1 ou 2, dans laquelle l'alcool polyvinylique a une masse moléculaire d'au moins 50.000.

4. Composition ou composant selon la revendication 3, dans lequel l'alcool polyvinylique a une masse moléculaire d'au moins 100.000.

5. Composition ou composant selon l'une des revendications précédentes, comprenant en outre un électrolyte sélectionné à partir du sel de citrate et/ou de carbonate.

6. Composition ou composant selon l'une des revendications précédentes, dans lequel l'alcool polyvinylique a un point de fusion d'au moins 70°C.

7. Utilisation d'un alcool polyvinylique soluble dans l'eau dans une composition ou dans un composant détergent granulaire comprenant un tensioactif non ionique et un édificateur de détergence afin de réduire ou d'éviter le

blocage de l'appareil permettant de produire ladite composition ou ledit composant, dans laquelle l'alcool polyvinylique ne représente pas plus de 5 % en masse de la composition détergente totale, et dans laquelle l'alcool polyvinylique est tel qu'un mélange de

- (1) une partie de 10 % en masse d'une solution aqueuse de l'alcool polyvinylique, et
 - (2) 2 parties de tensioactif non ionique comprenant un rapport de 1 pour 1 d'un éthoxylat d'alcool en C13 - C15 ayant en moyenne 3 unités d'éthoxylation et un éthoxylat d'alcool en C13 - C15 ayant en moyenne 6,5 unités d'éthoxylation,
- a une viscosité d'au moins 1,5 Pa.s à 40°C et un taux d'éthoxylation de 50 s⁻¹, et d'au moins 0,8 Pa.s à 60°C et un taux de cisaillement de 50 s⁻¹.

8. Utilisation d'un alcool polyvinylique tel que défini revendication 7, afin d'éviter ou de réduire le blocage dans une couche fluide fonctionnant à une température élevée.

9. Procédé de production d'une composition ou d'un composant détergent granulaire selon la revendication 1, dans lequel les granulés de composition ou de composant sont soumis dans une zone de séchage à une température supérieure à 45°C, ledit procédé comprenant les étapes consistant à incorporer l'alcool polyvinylique soluble dans l'eau dans la composition ou dans le composant comprenant l'agent tensioactif non ionique et un édificateur de détergence, de telle sorte que l'on réduit ou que l'on évite le blocage de la zone de séchage.