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(54) **PROCESS FOR PREPARING GRANULAR DETERGENT COMPOSITIONS**

VERFAHREN ZUR HERSTELLUNG GRANULARER WASCHMITTELZUSAMMENSETZUNGEN
PROCEDE RELATIF A L'ELABORATION DE COMPOSITIONS DETERGENTES GRANULEUSES

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Description**FIELD OF THE INVENTION**

5 **[0001]** The present invention relates to a process for preparing medium to low bulk density granular detergent compositions. More particularly, the invention is directed to a process in which a liquid binder is contacted with a solid starting material in a high-speed mixer and the resulting mixture treated in a medium- or low-speed mixer and finally in a gas fluidisation granulator, where more liquid binder is added.

BACKGROUND OF THE INVENTION

10 **[0002]** Recently, there has been considerable interest in the detergent industry to develop processes for the production of granulated detergent powders exhibiting specific bulk densities. Conventionally, detergent compositions have been produced by a spray-drying process in which the components of the composition are mixed with water to form an aqueous slurry which is then sprayed into a tower and contacted with hot air to remove water. The resulting spray-dried powders are highly porous and typically have a bulk density of 300 to 550 g/l.

15 **[0003]** Spray-dried powders generally provide good powder delivery characteristics such as dispensing and dissolution. However, the capital and operating costs of the spray-drying process are high. Nevertheless, there remains a significant consumer demand for such low density powders.

20 **[0004]** It is difficult to increase the bulk density of powders produced merely by spray-drying to much above 600 g/l without adversely effecting the performance of the detergent composition, e.g. high levels of sodium sulphate can be included in the slurry to increase the bulk density but such an ingredient does not contribute to detergency. Therefore, flexibility in the substantial bulk density can only be achieved by using additional "post-tower" processing steps which densify the powders.

25 **[0005]** In recent years, there has been much interest in the production of detergent products by processes which employ mainly mechanical mixing, without the use of spray-drying. In this type of process, the various components are dry-mixed and optionally granulated with a liquid binder. Liquid binders typically used in such granulation processes are anionic surfactants, acid precursors of anionic surfactants, nonionic surfactants, or any mixture thereof. Using these granulation processes, granular detergent products having a high bulk density, typically greater than 700 or 800 g/l, have been produced.

30 **[0006]** Granular products having a high bulk density have a low packing volume which is advantageous for storage and distribution operations and also for the consumer. Furthermore, if a spray-drying step is not employed, the capital and operating costs are typically much lower and the process uses less energy and so provides an environmental benefit.

35 **[0007]** However, such high density products typically have a much lower porosity than a conventional spray-dried powder which may impair the delivery of the product to the wash. For example, high bulk density, concentrated powders can have slow and/or incomplete dissolution in the wash liquor, leading to undissolved product residues on the laundry fabric. More particularly, zeolite-containing concentrated powders are known to have a problem with undissolved zeolite-containing powder getting trapped in the cloth fibre. This manifests itself as white specks, clearly visible and displeasing to the eye.

40 **[0008]** Attempts have been made to use mixing processes which do not employ a spray-drying step to produce lower bulk density materials. However, these tend to employ non-conventional detergent ingredients such as, for example, burkeite, which tend to be expensive and which therefore add to the cost of the product.

45 **[0009]** Consequently, the detergent industry has been looking for methods of producing granular products of medium to low bulk density, for example less than about 900 g/l, e.g. less than 800 g/l, preferably less than 750 g/l, that does not necessitate a spray-drying step. In particular there remains a need for a process for producing medium to low bulk density zeolite-containing powders, which does not employ a spray-drying step, which uses conventional starting ingredients and which yields a product with good powder properties.

PRIOR ART

50 **[0010]** Until recently, there has been less effort in developing the use of low-shear mixer/granulators such as, for example, gas fluidisation granulators.

55 **[0011]** WO98/58046, WO98/58047, WO98/58048 and WO99/00475 (Unilever) relate to low-shear granulation processes in which a liquid binder is sprayed onto a fluidising particulate material, preferably in a gas fluidisation granulator. They describe how the process parameters of gas fluidisation granulation can be controlled to produce desirable powder properties, e.g. particle size distribution, bulk density, flow properties and even the yield to some extent.

[0012] Low-shear granulation processes tend to give powders of lower bulk density, e.g. typically less than 650 g/l. In order to provide greater flexibility in bulk density, WO97/22685 (Unilever) discloses a process where a particulate starting

material is partially granulated with a liquid binder in a high- or moderate-speed mixer prior to a low-shear granulation step, for example in a fluidised bed, where further liquid binder is added and granulation completed.

5 [0013] WO98/58046, WO98/58047, WO98/58048 and WO99/00475 (Unilever) also disclose the use of a preceding partial granulation step as described in WO97/22685 (Unilever) in order to provide greater flexibility in bulk density. However, none of these references specifically describe or exemplify the use of more than one mixing/granulation step prior to the gas fluidisation granulation step.

10 [0014] WO98/14551, WO98/14552, WO98/14553, WO98/14556, WO98/14557 and WO98/14558 (Procter and Gamble) all relate to a non-tower process for preparing high bulk density granular detergent compositions. A surfactant, preferably in paste or liquid form is dispersed with a fine powder of diameter 0.1 to 500 microns in a first mixer under defined operating conditions and the agglomerates thus formed are then either further agglomerated in one or more mixing steps, the conditions of which are defined, and finally granulated in a fluidised bed under specified conditions. A clear distinction is made between the agglomerating action of the mixers and the granulating action of the fluid bed. The term "granulating" is defined as referring to fluidising agglomerates thoroughly for producing free flowing, round-shape granulated agglomerates. Therefore, although growth of agglomerates, for example through coating, may take place in the fluid bed, these cases clearly teach that agglomeration of the already formed agglomerates should not occur.

15 [0015] EP 264,049 (Bayer) describes a continuous process for the build-up granulation of organic substances, preferably dyes. The process involves granulating a pulverulent material in a high-speed mixer with a granulating liquid, further granulating in a moderate-speed mixer, preferably with the addition of further granulating liquid, and drying the product in a fluidised bed whilst simultaneously spraying with a forming auxiliary. In the final step, the material is preferably dried in a first part of the fluidised bed and then, in another section of the drier, sprayed with a "forming auxiliary" such as for example a sugar solution. In other words, the dried material is acting as a carrier and the forming auxiliary is absorbed onto the carrier, probably in the form of a coating. There is no teaching in EP 264,049 of further agglomeration occurring in the fluid bed.

20 [0016] WO99/03964, WO99/03966 and WO99/03967 (Procter & Gamble) describe a process for the making detergent powders of bulk density 300-550 g/l comprising agglomerating an anionic surfactant paste or acid precursor thereof with a dry starting material in a first high-speed mixer, mixing the detergent agglomerates in a second high-speed mixer to obtain built-up agglomerates, and further agglomerating the built-up agglomerates with a binder in a fluid bed drier and drying in the fluid bed drier. Low bulk densities are disclosed as being obtained by controlling the Stokes Number in the fluid bed agglomeration step, by controlling the nozzle height in the fluid bed agglomeration step or by controlling the particle size through all three of the steps.

25 [0017] We have found that a problem associated with the processes described in the prior art for producing medium to low bulk density powders involving combinations of mechanical mixing and gas fluidisation granulation steps is that the level of fines in the product powder is far from optimal. For example, the processes described in WO99/03964, WO99/03966 and WO99/03967 (Procter & Gamble) produce 14% fines (defined as being those particles of less than 150 microns) in the product. Indeed, a relatively high fines level would appear to be a preferred feature of these inventions. The fines are of the correct size to act as starting material for the process and are hence recycled back into the first high-speed mixer.

30 [0018] Surprisingly, we have found that powders of a medium to low bulk density and with improved powder properties can be produced in a process comprising contacting and mixing a liquid binder with a particulate starting material in a high-speed mixer, followed by mixing in a moderate- or low-speed mixer, and finally mixing with further liquid binder in a gas fluidisation granulator. More particularly, a significantly lower level of fines is obtained as compared with the prior art methods. Furthermore, the process produces powders with better flow properties, than those of the prior art.

45 DEFINITION OF THE INVENTION

[0019] The invention provides a process according to claim 1.

[0020] A granular detergent product of bulk density less than 900 g/l is obtained according to the process of the invention.

50 DETAILED DESCRIPTION OF THE INVENTION

Definitions

55 [0021] Hereinafter, in the context of this invention, the term "granular detergent product" encompasses granular finished products for sale, as well as granular components or adjuncts for forming finished products, e.g. by post-dosing to or with, or any other form of admixture with further components or adjuncts. Thus a granular detergent product as herein defined may, or may not contain detergent-active material such as synthetic surfactant and/or soap. The minimum requirement is that it should contain at least one material of a general kind of conventional component of granular detergent products, such as a surfactant (including soap), a builder, a bleach or bleach-system component, an enzyme,

an enzyme stabiliser or a component of an enzyme stabilising system, a soil anti-redeposition agent, a fluorescer or optical brightener, an anti-corrosion agent, an anti-foam material, a perfume or a colourant.

[0022] However, in a preferred embodiment of this invention granular detergent products contain detergent-active material such as synthetic surfactant and/or soap at a level of at least 5 wt%, preferably at least 10 wt% of the product.

[0023] As used hereinafter, the term "powder" refers to materials substantially consisting of grains of individual materials and mixtures of such grains. As used hereinafter, the term "granule" refers to a small particle of agglomerated smaller particles, for example, agglomerated powder particles. The final product of the process according to the present invention consists of, or comprises a high percentage of granules. However, additional granular and or powder materials may optionally be post-dosed to such a product.

[0024] "Fines", according to this invention, are defined as particles with a diameter of less than 180 microns.

[0025] "Coarse" material, according to this invention, is defined as those particles with a diameter greater than 1400 microns.

[0026] Levels of fine and coarse particles can be measured using sieve analysis.

[0027] For the purposes of this invention, the flow properties of the granular product are defined in terms of the dynamic flow rate (DFR), in ml/s, measured by means of the following procedure. A cylindrical glass tube of internal diameter of 35 mm and length of 600 mm is securely clamped with its longitudinal axis in the vertical position. Its lower end is terminated by a cone of polyvinyl chloride having an internal angle of 15° and a lower outlet orifice of diameter 22.5 mm. A first beam sensor is positioned 150 mm above the outlet, and a second beam sensor is positioned 250 mm above the first sensor.

[0028] To determine the dynamic flow rate, the outlet orifice is temporarily closed and the cylinder filled with the granular detergent product to a point about 10 cm above the upper sensor. The outlet is opened and the flow time t (seconds) taken for the powder level to fall from the upper sensor to the lower sensor measured electronically. This is repeated 2 or 3 times and an average time taken. If V is the volume (ml) of the tube between the upper and lower sensors, the DFR is given by V/t.

[0029] The Froude Number (Fr) is used to evaluate the relative effect of gravitational and centrifugal forces exerted on particles in a particular mixing device. As used in this invention, the Froude Number is defined as

$$Fr = \omega^2 d / 2g$$

Where ω = rotational speed of the agitator [rad/s]

d = diameter of the agitator [m]

g = acceleration due to gravity [m/s²]

[0030] Unless specified otherwise, values relating to powder properties such as bulk density, DFR, moisture content etc. relate to the weathered granular detergent product.

The Process

[0031] The process of this invention is carried out using a high-speed mixer, a moderate- or low-speed mixer and a gas fluidisation granulator.

The high- and moderate- or low-speed mixers:

Steps (i) and (ii)

[0032] The mixers used in the process of the invention, whether of high-, moderate- or low-speed, essentially consist of a hollow static cylinder or bowl, preferably horizontal, having a centrally mounted rotating shaft with tools mounted thereon.

[0033] The tools on the shaft of the high-speed mixer should provide a thorough, high-energy mixing action on the solids and the liquids which are being admixed at this stage. In this respect, the tools can, for example, be rod-like, shovel-like or a combination, or of any other appropriate design as will be well-known to the person skilled in the art.

[0034] Suitable high-speed mixers are any one of a variety of commercially available mixers such as, for example, those available from Lödige, Schugi and Drais. Particularly preferred machines include the Lödige (Trade mark) CB Recycler machine and the Drais (Trade Mark) K-TTP.

[0035] A suitable example of a moderate- or slow-speed mixer is a Lödige (Trade Mark) KM mixer, also referred to as Lödige Ploughshare. This apparatus has mounted on its shaft various plough-shaped tools. Optionally, one or more high-speed cutters can be used to prevent the formation of oversize or lumpy material. Another suitable machine for

this step is, for example the Drais (Trade Mark) K-T.

[0036] The minimum tip speed of the tools in the high-speed mixer is at least 10, and preferably at least 15 m/s. The maximum tip speed is preferably no greater than 60, more preferably no greater than 55, yet more preferably no greater than 50 and most preferably no greater than 45 m/s.

[0037] The high-speed mixer is preferably operated at a minimum Froude Number of at least 5, more preferably at least 20, yet more preferably at least 40 and most preferably at least 50. The maximum Froude Number is preferably no greater than 750, more preferably no greater than 500, and more preferably no greater than 400.

[0038] The maximum tip speed of the tools in the moderate- or low-speed mixer is less than 10, preferably less than 8, and most preferably less than 6 m/s. The minimum tip speed is preferably at least 1, more preferably at least 1.5 and most preferably at least 2 m/s.

[0039] The moderate- or low-speed mixer is preferably operated at a maximum Froude Number of less than 30, more preferably less than 15, yet more preferably less than 5, and most preferably less than 4. The minimum Froude Number is preferably at least 0.15, more preferably at least 0.30, yet more preferably at least 0.50, and most preferably at least 0.60.

[0040] It should be noted that in specifying any particular preferred tip speed or Froude Number range herein for either the high-speed mixer or the moderate- or low-speed mixer, no particular maximum tip speed is associated with any particular minimum tip speed. Likewise, no particular maximum Froude Number is associated with any particular minimum Froude Number.

[0041] The essential difference between the moderate- or low-speed mixer and the high-speed mixer in the process of this invention is that the moderate- or low-speed mixer operates at a lower tip speed and/or lower Froude number, preferably both.

[0042] The rotational speed of the shaft, with the tools mounted thereon, depends to a large degree on the scale of mixer being used. For example, a high-speed Lödige CB 100 machine will preferably have a shaft rotation speed in the range of from 100-1100 and more preferably from 200-750 rpm. Likewise, a moderate-speed Lödige KM 10000 machine will preferably have a shaft rotation speed in the range of from 20-200, more preferably from 25-120, yet more preferably from 30-100, and most preferably from 30-70 rpm. At other mixer scales, the preferred rotational speed is adjusted so as to maintain the tool tip speed and Froude number at the preferred levels given above.

[0043] In addition to the stirring/mixing action of the tools, the moderate- or low-speed mixer may contain cutters which can be driven independently of the shaft and tools. These cutters can be used to prevent the formation of lumpy or oversize material. If used, these are preferably operated at a rate of from 200-3000 and more preferably from 2000-3000 rpm.

[0044] The residence time in the mixers during steady state operation is dependent on parameters which include the rotational speed of the shaft, the throughput, the position of the tools and the weir at the exit opening. The residence time in the high-speed mixer should be relatively short, preferably from about 1 to 60 seconds, more preferably from 5-30 seconds, and yet more preferably from 5-20 seconds. The residence time in the moderate- or low-speed mixer should generally be longer than that in the high-speed mixer. Preferably it is in the range of from about 30 seconds to 10 minutes, more preferably from 30 seconds to 5 minutes, most preferably from 30 seconds to 3 minutes.

[0045] Other suitable machines which may be utilised in the process of this invention either as high-speed mixers or as moderate- or low-speed mixers include mixers of the Fukae^R FS-G series; Diosna^R V series ex Dierks & Sohne, Germany; Pharma Matrix^R ex T.K. Fielder Ltd; England; Fuji^R VG-C series ex Fuji Sangyo Co., Japan; the Roto^R ex Zanchetta & Co. srl, Italy and the Schugi^R Flexomix granulator.

[0046] The temperature in the high- and moderate- or low-speed mixers can be elevated and/or lowered by any appropriate means, e.g. a heating/cooling jacket.

[0047] The process in the mixers can be batch or continuous, however it is preferably continuous.

The gas fluidisation granulator : Step (iii)

[0048] The third step of the process of the invention utilises a gas fluidisation granulator. In this kind of apparatus, a gas (usually air) is blown through a body of particulate solids into or onto which is sprayed a liquid component. A gas fluidisation granulator is sometimes called a "fluidised bed" granulator or mixer. This is not strictly accurate since such mixers can be operated with a gas flow rate so high that a classical "bubbling" fluid bed does not form.

[0049] The gas fluidisation granulation and agglomeration process step is preferably carried out substantially as described in WO98/58046 and WO98/58047 (Unilever), the contents of which are hereby incorporated by way of Reference.

[0050] The gas fluidisation apparatus basically comprises a chamber in which a stream of gas (hereinafter referred to as the fluidisation gas), usually air, is used to cause turbulent flow of particulate solids to form a "cloud" of the solids and liquid binder is sprayed onto or into the cloud to contact the individual particles. As the process progresses, individual particles of solid starting materials become agglomerated, due to the liquid binder, to form granules.

[0051] The gas fluidisation granulator is typically operated at a superficial air velocity of about 0.1-1.2 ms⁻¹, either

under positive or negative relative pressure and with an air inlet temperature (ie fluidisation gas temperature) ranging from -10°C or 5°C up to 100°C. It may be as high as 200°C in some cases.

[0052] The fluidisation gas temperature, and thus preferably the bed temperature, may be changed during the granulation process as described in WO98/58048. It may be elevated for a first period, e.g. at up to 100°C or even up to 200°C and then at one or more other stages (before or after), it may be reduced to just above, at, or below ambient, e.g. to 30°C or less, preferably 25°C or less or even as low as 5°C or less or -10°C or less.

[0053] In a preferred embodiment, the fluidisation gas temperature, and preferably also the bed temperature, is elevated for a first period and subsequently lowered in a second period.

[0054] When the process is a batch process, the temperature variation will be effected over time. If it is a continuous process, it will be varied along the "track" of the granulator bed (i.e. in the direction of powder flow through the granulator bed). In the latter case, this is conveniently effected using a granulator of the "plug flow" type, i.e. one in which the materials flow through the reactor from beginning to end.

[0055] In a batch process, the fluidisation gas temperature may be reduced over a relatively short period of time, for example 10 to 50% of the process time. Typically, the gas temperature may be reduced for 0.5 to 15 minutes. In a continuous process, the gas temperature may be reduced along a relatively short length of the "track" of the granulator bed, for example along 10 to 50% of the track. In both cases, the gas may be pre-cooled.

[0056] Preferably, the fluidisation gas temperature, and preferably also the bed temperature, is not lowered until agglomeration of the fluidising particulate solid material is substantially complete.

[0057] In addition to the fluidisation gas, a gas fluidisation granulator may also employ an atomising gas stream. Such an atomising gas stream is used to aid atomisation of the liquid binder from the nozzle onto or into the fluidising solids. If an atomising gas stream is employed, it is generally operated at a pressure of from 2 to 5 bar. The atomising gas stream, usually air, may also be heated.

[0058] In a preferred embodiment, the bed temperature is maintained at around or near the pumpable temperature (as defined hereinbelow) of the liquid binder for at least part of the time and preferably for substantially the entire time the liquid binder is being sprayed onto the fluidising solids. This is especially preferred when the liquid binder is a structured blend (as described hereinbelow).

[0059] Alternatively, it is preferred that one, and preferably both of the fluidisation gas temperature and the atomisation gas temperature be elevated to a temperature which is within 15°C (plus or minus), and preferably within 10°C of the pumpable temperature of the liquid binder, especially when the liquid binder is a structured blend. The temperature should be elevated for at least part of, and preferably for substantially all of the period over which the liquid binder blend is being sprayed onto the fluidising material.

[0060] In a preferred embodiment, one, and preferably both of the fluidisation gas temperature and the atomisation gas temperature are elevated so as to be at least at the pumpable temperature of the liquid binder, especially when the liquid binder is a structured blend.

[0061] As used herein, the term "bed temperature" refers to the temperature of the fluidising gas around the solid particulate material. The bed temperature can be measured, for example, using a thermocouple probe. Whether there is a discernible powder bed or no discernible powder bed (ie because the mixer is being operated with a gas flow rate so high that a classical "bubbling" fluid bed is not formed), the "bed temperature" is taken to be the temperature as measured at a point inside the fluidisation chamber about 15 cm from the gas distributor plate.

[0062] The gas fluidisation granulator may optionally be of the kind provided with a vibrating bed, particularly for use in continuous mode.

Drying and/or cooling : Step (iv)

[0063] For use, handling and storage, the granular detergent product must be in a free flowing state. Therefore, in a final step, the granules can be dried and/or cooled if necessary. This step can be carried out in any known manner, for instance in a fluid bed apparatus (drying and cooling) or in an airlift (cooling). Drying and/or cooling can be carried out in the same fluid bed apparatus as used for the final agglomeration step simply by changing the process conditions employed as will be well-known to the person skilled in the art. For example, fluidisation can be continued for a period after addition of liquid binder has been completed and the air inlet temperature can be reduced.

[0064] In a preferred embodiment, the entire process is continuous.

The liquid binder

[0065] In the process of this invention, a liquid binder is added in steps (i) and (iii). Liquid binder may also be added in step (ii) of the process but it is preferred that little or no liquid is added. If liquid binder is added during step (ii), it is preferred that of the total amount of liquid binder added in the process, less than 10 wt%, preferably less than 5 wt% is added in step (ii).

[0066] The liquid binder added at each step may be the same or different and more than one liquid binder may be added in any one step.

[0067] The weight ratio of binder added in step (i) to that added in step (iii) is preferably in the range from 20:1 to 1:20, preferably from 10:1 to 1:10 and more preferably from 9:1 to 1:2. Preferably, of the total amount of liquid binder added in steps (i) and (iii), at least 5 wt%, more preferably at least 10 wt% is added in step (iii).

[0068] Generally, for the same process conditions, the higher the weight ratio of liquid binder added in step (i) to step (iii), the higher the resulting bulk density. Therefore, the bulk density of the granular detergent product can be varied and controlled to a certain degree by altering the ratio of binder addition.

[0069] The liquid binder may simply be pumped into the mixer of steps (i), and optionally the mixer of step (ii), or may be introduced as a spray. The liquid binder is sprayed into the gas fluidisation granulator of step (iii).

[0070] The liquid binder can comprise one or more components of the granular detergent product. Suitable liquid components include anionic surfactants and acid precursors thereof, nonionic surfactants, fatty acids, water and organic solvents.

[0071] The liquid binder can also comprise solid components dissolved in or dispersed in a liquid component, such as, for example, inorganic neutralising agents and detergency builders. The only limitation is that with or without dissolved or dispersed solids, the liquid binder should be pumpable and capable of being delivered to the mixer and/or granulator in a fluid, including paste-like, form.

[0072] It is preferred that the liquid binder comprises an anionic surfactant. The content of anionic surfactant in the liquid binder may be as high as possible, e.g. at least 98 wt% of the liquid binder, or it may be less than 75 wt%, less than 50 wt% or less than 25 wt%. It may, of course constitute 5 wt% or less or not be present at all.

[0073] Suitable anionic surfactants are well-known to those skilled in the art. Examples suitable for incorporation in the liquid binder 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.

[0074] It is very much preferred to form some or all of any anionic surfactant *in situ* in the liquid binder by reaction of an appropriate acid precursor and an alkaline material such as an alkali metal hydroxide, e.g. NaOH. Since the latter normally must be dosed as an aqueous solution, that inevitably incorporates some water. Moreover, the reaction of an alkali metal hydroxide and acid precursor also yields some water as a by-product.

[0075] However, in principle, any alkaline inorganic material can be used for the neutralisation but water-soluble alkaline inorganic materials are preferred. Another preferred material is sodium carbonate, alone or in combination with one or more other water-soluble inorganic materials, for example, sodium bicarbonate or silicate. If desired, a stoichiometric excess of neutralising agent may be employed to ensure complete neutralisation or to provide an alternative function, for example as a detergency builder, e.g. if the neutralising agent comprises sodium carbonate. Organic neutralising agents may also be employed.

[0076] Of course, if the liquid binder contains an acid precursor of an anionic surfactant, the acid precursor can be neutralised or neutralisation completed *in situ* in the mixer and/or granulator by either contacting with a solid alkaline material or adding a separate liquid neutralising agent to the mixer and/or granulator. However, neutralisation in the mixer and/or granulator is not a preferred feature of this invention.

[0077] The liquid acid precursor may be selected from linear alkyl benzene sulphonic (LAS) acids, alphaolefin sulphonic acids, internal olefin sulphonic acids, fatty acid ester sulphonic acids and combinations thereof. The process of the invention is especially useful for producing compositions comprising alkyl benzene sulphonates by reaction of the corresponding alkyl benzene sulphonic acid, for instance Dobanoic acid ex Shell. Linear or branched primary alkyl sulphates (PAS) having 10 to 15 carbon atoms can also be used.

[0078] In a preferred embodiment, the liquid binder comprises an anionic surfactant and a nonionic surfactant. The weight ratio of anionic surfactant to nonionic surfactant is in the range from 10:1 to 1:15, preferably from 10:1 to 1:10, more preferably 10:1 to 1:5. If the liquid binder comprises at least some acid precursor of an anionic surfactant and a nonionic surfactant, then the weight ratio of anionic surfactant, including the acid precursor, to nonionic surfactant can be higher, for example 15:1.

[0079] The nonionic surfactant component of the liquid binder may be any one or more liquid nonionics selected from primary and secondary alcohol ethoxylates, especially C₈-C₂₀ aliphatic alcohols ethoxylated with an average of from 1 to 20 moles 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, glycerol monoethers, and polyhydroxyamides (glucamide).

[0080] In a preferred embodiment the liquid binder is substantially non-aqueous. That is to say, the total amount of water therein is not more than 20 wt% of the liquid binder, preferably not more than 15 wt% and more preferably not more than 10 wt%. However, if desired, a controlled amount of water may be added to facilitate neutralisation. Typically, the water may be added in amounts of 0.5 to 2 wt% of the final detergent product. Typically, from 3 to 4 wt% of the liquid

binder may be water as the reaction by-product and the rest of the water present will be the solvent in which the alkaline material was dissolved. The liquid binder is very preferably devoid of all water other than that from the latter-mentioned sources, except perhaps for trace amounts/impurities.

[0081] Alternatively, an aqueous liquid binder may be employed. This is especially suited to manufacture of products which are adjuncts for subsequent admixture with other components to form a fully formulated detergent product. Such adjuncts will usually, apart from components resulting from the liquid binder, mainly consist of one, or a small number of components normally found in detergent compositions, e.g. a surfactant or a builder such as zeolite or sodium tripolyphosphate. However, this does not preclude use of aqueous liquid binders for granulation of substantially fully formulated products. In any event, typical aqueous liquid binders include aqueous solutions of alkali metal silicates, water soluble acrylic/maleic polymers (e.g. Sokalan CP5) and the like.

[0082] The liquid binder may optionally comprise dissolved solids and/or finely divided solids which are dispersed therein. The only limitation is that with or without dissolved or dispersed solids, the liquid binder should be pumpable and sprayable at temperatures of 50°C or greater or at any rate, 60°C or greater e.g. 75°C. Preferably it is solid at below 50°C, preferably at 25°C or less. The liquid binder is preferably at a temperature of at least 50°C, more preferably at least 60°C when fed into the mixer or gas fluidisation granulator.

[0083] According to the present invention, liquid binders are considered readily pumpable if they have a viscosity of no greater than 1 Pa.s at a shear rate of 50 s⁻¹ and at the temperature of pumping. Liquid binders of higher viscosity may still in principle be pumpable, but an upper limit of 1 Pa.s at a shear rate of 50 s⁻¹ is used herein to indicate easy pumpability.

[0084] The viscosity can be measured, for example, using a Haake VT500 rotational viscometer. The viscosity measurement may be carried out as follows. A SV2P measuring cell is connected to a thermostatic waterbath with a cooling unit. The bob of the measuring cell rotates at a shear rate of 50 s⁻¹. Solidified blend is heated in a microwave to 95°C and poured into the sample cup. After conditioning for 5 minutes at 98°C, the sample is cooled at a rate of +/- 1°C per minute. The temperature at which a viscosity of 1 Pa.s is observed, is recorded as the "pumpable temperature".

[0085] The "pumpable temperature" of the liquid binder is therefore defined herein as the temperature at which the liquid binder exhibits a viscosity of 1 Pa.s at 50 s⁻¹.

[0086] A definition of solid can be found in the Handbook of Chemistry and Physics, CRC Press, Boca Raton, Florida, 67th edition, 1986.

Structured blends

[0087] In a preferred embodiment of this invention, the liquid binder contains a structurant and liquid binders which contain a structurant are referred to herein as structured blends. All disclosures made herein with reference to liquid binders apply equally to structured blends.

[0088] In the context of the present invention, the term "structurant" means any component which enables the liquid component to achieve solidification in the granulator and hence good granulation, even if the solid component has a low liquid carrying capacity.

[0089] Structurants may be categorised as those believed to exert their structuring (solidifying) effect by one of the following mechanisms, namely: recrystallisation (e.g. silicate or phosphates); creation of a network of finely divided solid particles (e.g. silicas or clays); and those which exert steric effects at the molecular level (e.g. soaps or polymers) such as those types commonly used as detergency builders. One or more structurants may be used.

[0090] Structured blends provide the advantage that at lower ambient temperatures they solidify and as a result lend structure and strength to the particulate solids onto which they are sprayed. It is therefore important that the structured blend should be pumpable and sprayable at an elevated temperature, e.g. at a temperature of at least 50°C, preferably of at least 60°C, and yet should solidify at a temperature below 50°C, preferably below 35°C so as to impart its benefit.

[0091] Typically, in the high-speed and moderate- or low-speed mixers the temperature is more than 10°C, preferably more than 20°C below the temperature at which the blend is prepared and pumped into the granulator.

[0092] The structurants cause solidification in the liquid binder component preferably to produce a blend strength as follows. The strength (hardness) of the solidified liquid component can be measured using an Instron pressure apparatus.

A tablet of the solidified liquid component, taken from the process before it contacts the solid component, is formed of dimensions 14 mm in diameter and 19 mm in height. The tablet is then destroyed between a fixed and a moving plate, the moving plate moving towards the fixed plate. The speed of the moving plate is set to 5 mm/min, which causes a measuring time of about 2 seconds. The pressure curve is logged on a computer. Thus, the maximum pressure (at the moment of tablet breaking) is given and the E-modulus is calculated from the slope.

[0093] For the solidified liquid component, P_{max} at 20°C is preferably a minimum of 0.1 MPa, more preferably 0.2 MPa, e.g. from 0.3 to 0.7 MPa. At 55°C, a typical range is from 0.05 to 0.4 MPa. At 20°C, E_{mod} for the liquid blend is preferably a minimum of 3 MPa, e.g. from 5 to 10 MPa.

[0094] The structured blend is preferably prepared in a shear dynamic mixer for premixing the components thereof

and performing any neutralisation of anionic acid precursor.

[0095] Soaps represent one preferred class of structurant, especially when the structured blend comprises a liquid nonionic surfactant. In many cases it may be desirable for the soap to have an average chain length greater than the average chain length of the liquid nonionic surfactant but less than twice the average chain length of the latter.

[0096] It is very much preferred to form some or all of any soap structurant in situ in the liquid binder by reaction of an appropriate fatty acid precursor and an alkaline material such as an alkali metal hydroxide, e.g. NaOH. However, in principle, any alkaline inorganic material can be used for the neutralisation but water-soluble alkaline inorganic materials are preferred. In a liquid binder comprising an anionic surfactant and soap, it is preferred to form both the anionic surfactant and soap from their respective acid precursors. All disclosures made herein to formation of anionic surfactant by in situ neutralisation in the liquid binder of their acid precursors equally apply to the formation of soap in structured blends.

[0097] If desired, solid components may be dissolved or dispersed in the structured blend. Typical amounts of ingredients in the essential structured blend component as % by weight of the structured blend are as follows:

preferably from 98 to 10 wt% of anionic surfactant, more preferably from 70 to 30%, and especially from 50 to 30 wt%;

preferably from 10 to 98 wt% of nonionic surfactant, more preferably from 30 to 70 wt%, and especially from 30 to 50 wt%;

preferably from 2 to 30 wt% of structurant, more preferably from 2 to 20%, yet more preferably from 2 to 15 wt%, and especially from 2 to 10 wt%.

[0098] In addition to the anionic surfactant or precursor thereof, nonionic surfactant and structurant, the structured blend may also contain other organic solvents.

Solid starting material

[0099] The solid starting materials of this invention are particulate and may be powdered and/or granular. As such, the solid starting material may be any component of the granular detergent product that is available in particulate form. Preferably, the solid starting material with which the liquid binder is admixed comprises a detergency builder. In a particularly preferred embodiment of this invention, the solid starting material comprises builders selected from crystalline and amorphous aluminosilicates.

[0100] If the solid starting material comprises or substantially consists of an aluminosilicate builder, the weight ratio of liquid binder to the solid component is preferably from 0.2:1 to 0.8:1. If the solid component comprises or substantially consists of a phosphate builder, this ratio is preferably from 0.2:1 to 5:1.

Product

[0101] The present invention also encompasses a granular detergent product resulting from the process of the invention (before any post-dosing or the like).

[0102] Granular detergent products according to the invention have a bulk density of less than 900 g/l, preferably less than 800 g/l, more preferably less than 750 g/l, and yet more preferably less than 700 g/l. The bulk density may be as low as 450 g/l, however it is preferably greater than 550 g/l. Preferably it is in the range of 550-800 g/l, more preferably 550-750 g/l, yet more preferably 550-700 g/l.

[0103] The product will have a bulk density determined by the exact nature of the process but can be controlled to a certain degree by varying the ratio of addition of liquid binder in steps (i) and (iii).

[0104] The granular detergent products of the process of this invention are low in fines and possess good flow properties.

[0105] More particularly, the process of this invention provides granular detergent products with improved fines levels compared with similar powders made by the prior art methods. Preferably, not more than 10% by weight of the granules have a diameter of less than 180 microns, more preferably not more than 8% by weight. Furthermore, the granular detergent product preferably does not contain more than 10% by weight of granules of diameter greater than 1400 microns and more preferably, not more than 5% by weight of the granules are above this limit.

[0106] The granular product is considered to be free flowing if it has a DFR of at least 80 ml/s. Preferably the granular products of this invention have DFR values of at least 80 ml/s, preferably at least 90 ml/s, more preferably at least 100 ml/s, and most preferably at least 110 ml/s. These DFR values apply to the weathered product and preferably also to the unweathered product.

[0107] Finally, the granules may be distinguished from granules produced by other methods by using mercury porosimetry. The latter technique is ideal for characterising granules that have been prepared by a process involving gas

fluidisation agglomeration.

Detergent compositions and ingredients

5 **[0108]** As previously indicated, a granular detergent product prepared by the process of the invention may itself be a fully formulated detergent composition, or may be a component or adjunct which forms only a part of such a composition. This section relates to final, fully formed detergent compositions.

[0109] The total amount of detergency builder in the final detergent composition is suitably from 10 to 80 wt%, preferably from 15 to 60 wt%. The builder may be present in an adjunct with other components or, if desired, separate builder particles containing one or more builder materials may be employed.

10 **[0110]** This invention is especially applicable to use where the solid starting material comprises builders selected from crystalline and amorphous aluminosilicates, for example zeolites as disclosed in GB-A-1 473 201; amorphous aluminosilicates as disclosed in GB-A-1 473 202; and mixed crystalline/amorphous aluminosilicates as disclosed in GB 1 470 250; and layered silicates as disclosed in EP-B-164 514.

15 **[0111]** Aluminosilicates, whether used as layering agents and/or incorporated in the bulk of the particles may suitably be present in a total amount of from 10 to 60 wt% and preferably an amount of from 15 to 50 wt% based on the final detergent composition. 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-A-384 070 may be used. Zeolite MAP is an alkali metal aluminosilicated of the P type having a silicone to aluminium ratio not exceeding 1.33, preferably not exceeding 1.15, and more preferably not exceeding 1.07.

20 **[0112]** Other suitable builders include hydratable salts, preferably in substantial amounts such as at least 25% by weight of the solid component, preferably at least 10% by weight. Hydratable solids include inorganic sulphates and carbonates, as well as inorganic phosphate builders, for example, sodium orthophosphate, pyrophosphate and tripolyphosphate.

25 **[0113]** Other inorganic builders that may be present include sodium carbonate (as mentioned above, an example of a hydratable solid), if desired in combination with a crystallisation seed for calcium carbonate as disclosed in GB-A-1 437 950. As mentioned above, such sodium carbonate may be the residue of an inorganic alkaline neutralising agent used to form an anionic surfactant in situ.

30 **[0114]** 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, aminopolycarboxylates such as nitrilotriacetates (NTA), ethylenediaminetetraacetate (EDTA) and iminodiacetates, alkyl- and alkenylmalonates and succinates; and sulphonated fatty acid salts. A copolymer of maleic acid, acrylic acid and vinyl acetate is especially preferred as it is biodegradable and thus environmentally desirable. This list is not intended to be exhaustive.

35 **[0115]** Especially preferred organic builders are citrates, suitably used in amounts of from 2 to 30 wt%, preferably from 5 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%. The builder is preferably present in alkali metal salt, especially sodium salt, form.

40 **[0116]** The granular detergent compositions may contain, in addition to any anionic and/or nonionic surfactants of the liquid binder, one or more other detergent-active compounds which may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic surfactants, and mixtures thereof. These may be dosed at any appropriate stage before or during the process. 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. The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds.

45 **[0117]** The detergent compositions may also contain a bleach system, desirably a peroxy bleach compound, for example, an inorganic persalt or organic peroxyacid, capable of yielding hydrogen peroxide in aqueous solution. The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. An especially preferred bleach system comprises a peroxy bleach compound (preferably sodium percarbonate) optionally together with a bleach activator.

50 **[0118]** Usually, any bleach and other sensitive ingredients, such as enzymes and perfumes, will be post-dosed after granulation along with other minor ingredients.

55 **[0119]** Typical minor ingredients include sodium silicate; corrosion inhibitors including silicates; 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.

[0120] Optionally, a "layering agent" or "flow aid" may be introduced at any appropriate stage in the process of the invention. This is to improve the granularity of the product, e.g. by preventing aggregation and/or caking of the granules.

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Any layering agent flow aid is suitably present in an amount of 0.1 to 15 wt% of the granular product and more preferably in an amount of 0.5 to 5 wt%.

[0121] Suitable layering agents/flow aids include crystalline or amorphous alkali metal silicates, aluminosilicates including zeolites, citrates, Dicalol, calcite, diatomaceous earths, silica, for example precipitated silica, chlorides such as sodium chloride, sulphates such as magnesium sulphate, carbonates such as calcium carbonate and phosphates such as sodium tripolyphosphate. Mixtures of these materials may be employed as desired.

[0122] Zeolite MAP, as well as being a preferred builder, is especially useful as a layering agent. Layered silicates such as SKS-6 ex Clariant are also useful as layering agents.

Powder flow may also be improved by the incorporation of a small amount of an additional powder structurant, for example, a fatty acid (or fatty acid soap), a sugar, an acrylate or acrylate/maleate polymer, or sodium silicate which is suitably present in an amount of from 1 to 5 wt%.

[0123] In general, additional components may be included in the liquid binder or admixed with the solid starting material at an appropriate stage of the process. However, solid components can be post-dosed to the granular detergent product.

[0124] The granular detergent composition may also comprise a particulate filler (or any other component which does not contribute to the wash process) which suitably comprises an inorganic salt, for example sodium sulphate and sodium chloride. The filler may be present at a level of 5 to 70 wt% of the granular product.

[0125] The invention will now be described in more detail by way of the following non-limiting Examples, in which parts and percentages are by weight unless otherwise stated. Examples denoted by a number are in accordance with the invention, while those denoted by a letter are comparative.

EXAMPLES

EXAMPLES 1 to 6, COMPARATIVE EXAMPLES A and B

[0126] Granular detergent product base powders of the formulations detailed in Table 1 were prepared.

[0127] The base powders of Examples 1 to 6 were prepared by

(i) mixing and granulating solid starting materials consisting of zeolite, light soda ash, sodium carboxymethylcellulose (SCMC) and citrate with liquid binder in a Lödige Recycler (CB 30)

(ii) transferring the material from the Recycler to a Lödige Lödige Ploughshare (KM 300) mixer

(iii) transferring the material from the Ploughshare to a Vometec (Trade mark) fluid bed operating as a gas fluidisation granulator, adding further liquid binder and agglomerating, and

(iv) finally drying/cooling the product in the fluid bed.

[0128] The conditions in steps (i) to (iii) were as follows:

(i) Lödige Recycler (CB 30)

Residence time	about 15 seconds
Shaft rotation speed	1000 rpm
Tip speed	15.7 m/s
Froude number	168

(ii) Lödige Ploughshare (KM 300)

Residence time	about 3 minutes
Shaft rotation speed	100 rpm
Choppers	Switched off
Tip speed	2.62 m/s
Froude number	2.8
Liquid binder	None added

(iii) Fluid bed (Batch Vometec apparatus, batch size 10 kg:)

Superficial air velocity	1.0 m/s
FLuidisation gas temperature	75°C

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(continued)

Atomisation gas temperature	Hot
Atomisation air pressure	3.5 bar
Height of nozzle (above distributor plate)	47 cm
Rate of spray-on of binder	800 g/min

[0129] The base powders of comparative Examples A and B were prepared in the same way except that step (ii) was omitted.

[0130] The liquid binder used in steps (i) and (iii) was a structured blend comprising the anionic surfactant, nonionic surfactant and soap components of the base powder. The blend was prepared by mixing 38.44 parts by weight of LAS acid precursor and 5.20 parts by weight fatty acid precursor of the soap in the presence of 41.60 parts by weight nonionic surfactant in a blend-loop and neutralising with 14.75 parts of a sodium hydroxide solution. The blend temperature in the loop was controlled by a heat-exchanger. The neutralising agent was a sodium hydroxide solution. The resulting blend had the following composition :

	%
Sodium linear alkylbenzene sulphonate	39.9
Nonionic surfactant (7EO)	41.6
Soap	5.6
Water	12.9

[0131] The weight ratio of blend added in the recycler and gas fluidisation granulator was varied as detailed in Table 1.

[0132] The bulk density and DFR values for both the fresh and weathered product are given in Table 1, as are the levels of fine and coarse material in the product.

[0133] The DFR of both the weathered and fresh granular detergent products of Examples 1-6 was at least 100 ml/s. In fact the lowest DFR observed was in Example 1 at 108 ml/s.

[0134] The fines level in all of Examples 1 to 6 was less than 10 wt%.

[0135] The products of Comparative Examples A and B were prepared by the same process as used in Examples 1 and 2 respectively except that the second step in the moderate-speed mixer had been omitted in Examples A and B. Comparing Example A with Example 1 and Example B with Example 2, there is a clear benefit seen in employing the moderate-speed mixer (ie step (ii)) in terms of better DFR values (for both fresh and weathered product) and reduced levels of fines in the granular detergent products.

[0136] The results in Table 1 also clearly demonstrate a general decrease in bulk density of the product as the ratio of binder added in step (i) to that added in step (ii) decreases. A bulk density range of 739 to 579 g/l was observed.

Table 1

EXAMPLE	1	A	2	B	3	4	5	6
Base powder								
Na-LAS	11.35	11.35	11.66	11.66	12.08	12.23	12.77	13.30
Nonionic 7EO	11.72	11.72	12.04	12.04	12.47	12.63	13.19	13.73
Soap	1.58	1.58	1.62	1.62	1.68	1.70	1.78	1.85
Zeolite A24	37.47	37.47	37.07	37.07	36.53	36.32	35.63	34.95
Light soda-ash	25.90	25.90	25.63	25.63	25.25	25.12	24.64	24.17
SCMC	0.84	0.84	0.83	0.83	0.82	0.81	0.80	0.78
Citrate	3.45	3.45	3.41	3.41	3.36	3.35	3.28	3.22
Moisture, salts, NDOM	7.69	7.69	7.74	7.74	7.81	7.84	7.91	8.00
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

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(continued)

	Processing conditions								
5	Blend in recycler (%)	80	80	78	78	74	68	55	40
	Blend in fluid bed (%)	20	20	22	22	26	32	45	60
	Ploughshare used	Yes	No	Yes	No	Yes	Yes	Yes	Yes
	Fresh properties								
10	BD (g/l)	740	665	703	685	712	639	612	571
	DFR (ml/s)	108	100	115	100	122	123	125	115
	Weathered properties								
15	BD (g/l)	739	674	719	690	658	655	615	579
	DFR (ml/s)	115	85	110	95	122	130	120	112
	RRd	626	442	546	561	496	519	524	557
	RRn	1.77	1.7	1.96	1.66	2.07	2.21	2.55	2.39
20	Fines (<180) (%)	8.3	17.5	8.6	11.7	9.1	6.7	4.2	4.2
	Coarse (>1400) (%)	2.6	0.4	1.5	2.3	1	0.9	1	1.8

Claims

1. A process for preparing a granular detergent product comprising the steps of:
 - (i) mixing and agglomerating a liquid binder with a solid starting material in a high-speed mixer;
 - (ii) mixing the material from step (i) in a moderate- or low-speed mixer;
 - (iii) feeding the material from step (ii) and a liquid binder, which comprises one or more anionic surfactants or acid precursors thereof, into a gas fluidisation granulator and further agglomerating, and
 - (iv) optionally, drying and/or cooling,

in which the high-speed mixer operates with tool tip speed of at least 10 and preferably at least 15 m/s and the moderate- or low-speed mixer operates with a tool tip speed of less than 10, preferably less than 8 and more preferably less than 6 m/s.
2. The process according to claim 1 in which the high-speed mixer operates at a Froude Number of at least 5, preferably at least 20, more preferably at least 40 and yet more preferably at least 50.
3. The process according to any preceding claim, in which the moderate- or low-speed mixer operates at a Froude Number of less than 30, preferably less than 15, more preferably less than 8, more preferably less than 5, and yet more preferably less than 4.
4. The process according to any preceding claim, in which less than 10 wt% of the total liquid binder added in the process is added in step (ii).
5. The process according to claim 4, in which no liquid binder is added in step (ii).
6. The process according to any of one of the preceding claims, in which the weight ratio of binder added in step (i) to that added in step (iii) is in the range from 20:1 to 1:20, preferably from 10:1 to 1:10.
7. The process according to any preceding claim, in which the liquid binder in step (i) comprises one or more anionic surfactants or acid precursors thereof.
8. The process according to any preceding claim, in which the liquid binder comprises one or more nonionic surfactants.

9. The process according to any preceding claim, wherein the liquid binder is at a temperature of at least 50°C when fed into the mixer or gas fluidisation granulator.
10. The process according to any one of the preceding claims, in which the liquid binder is a structured blend.
11. The process according to claim 10, in which the structured blend contains a soap structurant.
12. The process according to any preceding claim, in which the liquid binder added in step (iii) is a structured blend, and the fluidising gas temperature and/or the atomisation gas temperature of the gas fluidisation granulator is elevated, as the structured blend is being added, to a temperature within 35°C, preferably within 25°C and more preferably within 15°C, of the pumpable temperature of the structured blend.
13. The process according to claim 12, in which the fluidising gas temperature and/or the atomisation gas temperature is around the pumpable temperature of the structured blend.
14. The process according to any preceding claim, in which the solid starting material comprises an aluminosilicate builder.
15. The process according to claim 14, in which the aluminosilicate builder comprises zeolite MAP.

Patentansprüche

1. Verfahren zur Herstellung eines granulären Waschmittelproduktes, umfassend die Schritte:

- (i) Mischen und Agglomerieren eines flüssigen Bindemittels mit einem festen Ausgangsmaterial in einem Hochgeschwindigkeitsmischer;
- (ii) Mischen des Materials aus Schritt (i) in einem Mischer mit mittlerer oder niedriger Geschwindigkeit;
- (iii) Einspeisen des Materials aus Schritt (ii) und eines flüssigen Bindemittels, das ein oder mehrere anionische oberflächenaktive Mittel oder einen Säurepräkursor davon umfasst, in einen Gaswirbelschichtgranulator und weiteres Agglomerieren, und
- (iv) gegebenenfalls Trocknen und/oder Kühlen,

wobei der Hochgeschwindigkeitsmischer mit einer Werkzeugschneidgeschwindigkeit von mindestens 10 und bevorzugt mindestens 15 m/s betrieben wird und der mittelschnelle oder langsame Mischer mit einer Werkzeugschneidgeschwindigkeit von weniger als 10, bevorzugt weniger als 8 und stärker bevorzugt weniger als 6 m/s betrieben wird.

2. Verfahren nach Anspruch 1 wobei der Hochgeschwindigkeitsmischer bei einer Froude-Zahl von mindestens 5, vorzugsweise mindestens 20, stärker bevorzugt mindestens 40 und noch stärker bevorzugt mindestens 50 betrieben wird.
3. Verfahren nach einem vorhergehenden Anspruch, wobei der mittelschnelle oder langsame Mischer bei einer Froude-Zahl von weniger als 30, vorzugsweise weniger als 15, stärker bevorzugt weniger als 8, stärker bevorzugt weniger als 5 und noch stärker bevorzugt weniger als 4 betrieben wird.
4. Verfahren nach einem vorhergehenden Anspruch, wobei weniger als 10 Gew.-% des gesamten flüssigen Bindemittels, das in dem Verfahren zugegeben wird, in Schritt (ii) zugegeben wird.
5. Verfahren nach Anspruch 4, wobei kein flüssiges Bindemittel in Schritt (ii) zugegeben wird.
6. Verfahren nach einem der vorhergehenden Ansprüche, wobei das Gewichtsverhältnis des Bindemittels, das in Schritt (i) zugegeben wird, zu dem, das in Schritt (iii) zugegeben wird, in dem Bereich von 20 : 1 bis 1 : 20, vorzugsweise 10 : 1 bis 1 : 10 liegt.
7. Verfahren nach einem vorhergehenden Anspruch, wobei das flüssige Bindemittel in Schritt (i) ein oder mehrere anionische oberflächenaktive Mittel oder einen Säurepräkursor davon umfasst.
8. Verfahren nach einem vorhergehenden Anspruch, wobei das flüssige Bindemittel einen oder mehrere nicht-ionische

oberflächenaktive Mittel umfaßt.

5 9. Verfahren nach einem vorhergehenden Anspruch, wobei das flüssige Bindemittel eine Temperatur von mindestens 50 °C besitzt, wenn es in den Mischer oder Gaswirbelschichtgranulator eingespeist wird.

10 10. Verfahren nach einem der vorhergehenden Ansprüche, wobei das flüssige Bindemittel eine strukturierte Mischung ist.

11. Verfahren nach Anspruch 10, wobei die strukturierte Mischung ein Seifenstrukturierungsmittel enthält.

12. Verfahren nach einem vorhergehenden Anspruch, wobei das flüssige Bindemittel, das in Schritt (iii) zugegeben wurde, eine strukturierte Mischung ist, und die Wirbelgastemperatur und/oder die Zerstäubungsgastemperatur des Gaswirbelschichtgranulators, wenn die strukturierte Mischung zugegeben wird, auf eine Temperatur innerhalb 35 °C, vorzugsweise innerhalb 25 °C und stärker bevorzugt innerhalb 15 °C, der pumpfähigen Temperatur der strukturierten Mischung erhöht wird.

13. Verfahren nach Anspruch 12, wobei die Wirbelgastemperatur und/oder die Zerstäubungsgastemperatur um die pumpfähige Temperatur der strukturierten Mischung liegt.

20 14. Verfahren nach einem vorhergehenden Anspruch, wobei das feste Ausgangsmaterial einen Alumosilikataufbaustoff umfaßt.

15. Verfahren nach Anspruch 14, wobei der Alumosilikataufbaustoff Zeolith MAP umfaßt.

25 Revendications

1. Procédé permettant de préparer un détergent granulaire comprenant les étapes consistant à :

- 30 (i) mélanger et agglomérer un liant liquide avec une matière première à l'état solide dans un malaxeur rapide ;
(ii) mélanger le matériau de l'étape (i) dans un malaxeur à vitesse lente ou à vitesse moyenne ;
(iii) introduire le matériau de l'étape (ii) et un liant liquide, qui comprend un ou plusieurs tensioactifs anioniques ou précurseurs acides de ceux-ci, dans un granulater à gaz fluidisant et agglomérer de nouveau, et
35 (iv) éventuellement sécher et/ou refroidir,

dans lequel le malaxeur rapide fonctionne à une vitesse périphérique d'au moins 10, et de préférence d'au moins 15 m/s, et le malaxeur à vitesse lente ou à vitesse moyenne fonctionne à une vitesse périphérique inférieure à 10, de préférence inférieure à 8, et mieux encore inférieure à 6 m/s.

40 2. Procédé selon la revendication 1, dans lequel le malaxeur rapide fonctionne à un nombre de Froude d'au moins 5, de préférence d'au moins 20, mieux d'au moins 40, et mieux encore d'au moins 50.

3. Procédé selon l'une quelconque des revendications précédentes, dans lequel le malaxeur à vitesse lente ou à vitesse moyenne fonctionne à un nombre de Froude inférieur à 30, de préférence inférieur à 15, mieux inférieur à 8, mieux encore inférieur à 5, et de manière préférée entre toutes inférieur à 4.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel on ajoute une proportion inférieure à 10% en poids du liant liquide total ajouté dans le procédé, dans l'étape (ii).

50 5. Procédé selon la revendication 4, dans lequel aucun liant liquide n'est ajouté dans l'étape (ii).

6. Procédé selon l'une quelconque des revendications précédentes, dans lequel le rapport en poids entre le liant ajouté dans l'étape (i) et celui ajouté dans l'étape (iii) se situe dans l'intervalle de 20/1 à 1/20, de préférence de 10/1 à 1/10.

55 7. Procédé selon l'une quelconque des revendications précédentes, dans lequel le liant liquide dans l'étape (i) comprend un ou plusieurs tensioactifs anioniques ou précurseurs acides de ceux-ci.

8. Procédé selon l'une quelconque des revendications précédentes, dans lequel le liant liquide comprend un ou plu-

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sieurs tensioactifs non ioniques.

5 9. Procédé selon l'une quelconque des revendications précédentes, dans lequel le liant liquide se trouve à une température d'au moins 50°C lorsqu'il est alimenté dans le malaxeur ou dans le granulateur à gaz fluidisant.

10. Procédé selon l'une quelconque des revendications précédentes, dans lequel le liant liquide est un mélange structuré.

11. Procédé selon la revendication 10, dans lequel le mélange structuré contient un agent structurant à base de savon.

10 12. Procédé selon l'une quelconque des revendications précédentes, dans lequel le liant liquide ajouté dans l'étape (iii) est un mélange structuré, et la température du gaz de fluidisation et/ou la température du gaz de pulvérisation du granulateur à gaz fluidisant est élevée étant donné que le mélange structuré est ajouté à une température égale, à plus ou moins 35°C près, de préférence à plus ou moins 25°C près, et mieux à plus ou moins 15°C près, à la température pompable du mélange structuré.

15 13. Procédé selon la revendication 12, dans lequel la température du gaz de fluidisation et/ ou la température du gaz de pulvérisation est voisine de la température pompable du mélange structuré.

20 14. Procédé selon l'une quelconque des revendications précédentes, dans lequel la matière première à l'état solide comprend un adjuvant à base d'aluminosilicate.

15. Procédé selon la revendication 14, dans lequel l'adjuvant à base d'aluminosilicate comprend la zéolite MAP.

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REFERENCES CITED IN THE DESCRIPTION

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