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(54) PROCESS FOR THE PRODUCTION OF A DETERGENT COMPOSITION

VERFAHREN ZUR HERSTELLUNG EINER WASCHMITTELZUSAMMENSETZUNG

PROCEDE DE PRODUCTION D'UNE COMPOSITION DETERGENTE

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EP-A- 0 367 339	**EP-A- 0 544 365**
EP-B- 0 270 240	**WO-A-97/02338**

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Description

Technical field

[0001] The present invention relates to a process for the production of a detergent composition. In particular the invention is concerned with a process for the production of a detergent composition having a medium or low bulk density.

Background to the invention

[0002] 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 crutcher slurry which is then sprayed into a spray-drying tower and contacted with hot air to remove water whereby detergent particles, often referred to as a "base" powder are obtained. The particles so obtained, have a high porosity. Thus powders produced by this method typically have a bulk density of 300 to 550 g/l or even up to 650 g/l.

[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.

[0004] In recent years, detergent powders having a high bulk density have been produced by mechanical mixing processes. Bulk densities of 700 to 900 g/l and even higher have been obtained. Typically such powders are produced by densifying a spray-dried base powder in one or more mechanical mixers, optionally with the addition of further components, or by mixing the components of the composition in a continuous or batch mixing process without the use of a spray-drying step.

[0005] Powders 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. The avoidance of a spray-drying step in the detergent production process is therefore often desirable.

[0006] However, such high density powders typically have a much lower porosity than a conventional spray-dried powderwhich may impair the delivery of the powder into the wash liquor. Additionally, the production of powders having a low to medium bulk density, for example less than about 700 g/l, has not hitherto been readily achievable on a commercial scale without the use of a spray-drying step.

[0007] EP-A-367 339 discloses a process for the production of a detergent composition having a high bulk density in which a particulate starting material is treated in a high speed mixer, a moderate speed mixer wherein the material is brought into or maintained in a deformable state, and then dried and/or cooled. The starting material may be a spray-dried base powder or the components of the composition may be employed without a prior spray-drying step in the detergent production process.

[0008] WO-A-97/02338 (Unilever : unpublished at the priority date of the present application) discloses that a low bulk density, for example less than 700 g/l, may be obtained by a process in which a spray-drying step is not employed, if the composition is formulated with a component having a low bulk density. However, this process is relatively unsuitable for use with starting materials which are either available commercially in a form in which the particle density is high or which are themselves produced by spray-drying (the latter normally producing relatively porous particles).

[0009] EP-A-544 365 discloses granulation of porous spray-dried detergent free starting material of 300 micron particle size In a "recycler" high speed mixer/densifier with a liquid binder comprising a primary alcohol sulphate anionic surfactant, a nonionic surfactant and water.

[0010] We have now found that medium or low bulk density powders may be obtained by a new process of mechanical mixing of a powder which contains little or no detergent active material and which consists of particles having a predetermined average particle size and high particle porosity together with a liquid component comprising a detergent active material or a precursor therefor.

Definition of the Invention

[0011] Thus, a first aspect of the present invention provides a process for the production of a detergent composition having a bulk density of no more than 750 g/l), e.g. no more than 700 or 650 g/l, the process comprising mixing a particulate starting material which contains no more than 10% by weight of the starting material of detergent active material and which starting material has a d_{50} average particle diameter of from 100μm to 1000μm and a particle porosity of at least 0.4, together with a liquid component comprising a detergent active material or a precursor therefor in a mixer/granulator having both a stirring and a cutting action, the stirrer is operated at a rate of 25 to 250 rpm and the cutter is operated at a rate of 300 to 3000 rpm.

Detailed description of the invention

[0012] The present invention derives from the unexpected observation that the bulk density of the resultant product is dependent upon the rotational speed of mixing. This is also a function of the particular mixer of choice but essentially, the lower the speed of the mixer, the lower the bulk density of the product.

[0013] This new process has two distinct but separate advantages. The first advantage is that by choosing a powder starting material which already possesses the required average particle size and porosity medium or low bulk density powders may be prepared.

[0014] The second advantage is obtainable in manufacturing scenarios where both spray-drying and mechanical mixture agglomeration facilities are available. By affording the possibility of using a spray-dried product as a starting material in a mechanical agglomeration process, the present invention provides a further degree of flexibility in such a modular approach to the production of detergent powder products. As used herein, the abbreviation "NTR" means "non-tower route", i.e. a powder produced by mixing rather than in a spray-drying tower even if the starting materials are themselves produced by spray drying.

[0015] Suitably, the detergent composition resulting from the process of the present invention has a bulk density of 400 to 650 g/l, preferably 450 to 650 g/l and more preferably 500 to 600 g/l. It is further preferred that the resultant detergent composition has a particle porosity of at least 0.2 and more preferably at least 0.25.

[0016] Suitably, the particulate starting material is dosed at a level of from 10 to 75 wt%, preferably from 20 to 40 wt%, of the composition resulting from the mechanical mixing process.

[0017] Instead of expressing particle size distributions in terms of average (e.g. d_{50}) particle diameters, if they are capable of being fitted to a Rosin-Rammler distribution, they may be expressed in terms of their Rosin Rammler number. This is calculated by fitting the particle size distribution to an n-power distribution according to the following formula:-

$$R = 100 * Exp\left\{-\left(\frac{D}{D_r}\right)^n\right\}$$

where R is the cumulative percentage of powder above a certain size D. D_r is the average granule size and n is a measure of the particle size distribution. D_r and n are the Rosin Rammler fits to a measured particle size distribution. A high n value means narrow particle size distribution and low values mean a broad particle size distribution.

[0018] The process may be a continuous process or may be performed batch-wise.

[0019] A suitable type of mixer/granulator for use in the process of the invention is bowl-shaped and preferably has a substantially vertical stirrer axis. Especially preferred are mixers of the Fukae (Trade Mark) FSOG series manufactured by Fukae Powtech Kogyo Co., Japan; this apparatus is essentially in the form of a bowl-shaped vessel accessible via a top part, provided near its base with a stirrer having a substantially vertical axis, and a cutter positioned on a side wall. The stirrer and cutter may be operated independently of one another, and at separately variable speeds.

[0020] Other similar mixers found to be suitable for use In the process of the invention are the Diosna (Trade Mark) V series ex Dierks & Sohne, Germany; and the Pharma Matrix (Trade Mark) ex T K Fielder Ltd., England. Other similar mixers suitable for use In the process of the invention include the Fuji (Trade Mark) VG-C series ex Fuji Sangyo Co., Japan; and the Roto (Trade Mark) ex Zanchetta & Co sri, Italy.

[0021] Granulation is effected by running the mixer using both stirrer and cutter; a relatively short residence time (for example, 5-8 minutes for a 35kg batch) is generally sufficient. The final bulk density can be controlled by choice of residence time and stirrer rate.

[0022] Suitably the stirrer is operated at a rate of 25 to 250 rpm, e.g. from 100 rpm to 200 rpm or even as low as 30 to 50 rpm. However, this speed is dependent on the size of the apparatus. Independently the cutter is suitably operated at a rate of 300 to 3000 rpm. For example, 300 to 2200 rpm. A batch process typically involves pre-mixing of solid components, addition of liquids, granulation, optional addition of a layering material suitable for controlling the granulation end-point, and product discharge. The rate of stirring and/or cutting is suitably adjusted according to the stage of the process. The mixing step is preferably carried out at a controlled temperature somewhat above ambient, preferably above 30°C. Suitably the temperature is within the range 30 to 45°C.

[0023] The amount of detergent active material in the particulate starting material is no more than 10% by weight of that material. However, the amount of detergent active material in the particulate starting material is suitably no more than 5% by weight thereof and preferably no more than 1% by weight thereof. The particulate starting material may be substantially or totally free of any detergent active material. Suitably, the particulate starting material may be one prepared by spray-drying. However, starting materials having the required parameters maybe obtained by other means, e.g. involving granulation.

[0024] The d_{50} average particle diameter of the particulate starting material is from 100μm to 1000μm. This is important for controlling the particle size distribution in the final product. Preferably though, this average particle diameter is from 150μm to 800μm, especially from 200μm to 700μm. Preferably, 90% by weight of the particles in the starting material have a particulate diameter in the region of 100μm to 1000μm.

[0025] The particle porosity of the particulate starting material is at least 0.4 but is preferably at least 0.45, e.g. from 0.45 to 0.55. Most preferably it is at least 0.50. In any event, such particulate starting material may comprise a spray-dried material, that is to say some or all of the starting material is formed by a spray-drying process.

[0026] The measurement of- particle porosity is based on the well known Kozeny-Carman relation for air flow through a packed bed of powder:

$$\frac{\phi_v h}{\Delta P} = k \frac{\pi D^2_{bed}}{4\eta} D_p^{\ 2} \frac{\varepsilon^3_{bed}}{(1-\varepsilon_{bed})^2}$$

In which:

ϕ_v=	airflow
ΔP =	pressure drop over the bed
D_{bed} =	bed diameter
h =	bed height
D_p =	particle diameter
ε_{bed} =	bed porosity
η =	gas viscosity
k =	empirical constant, equal to 180 for granular solids

[0027] The bulk density of a powder can be described by the following equation:

$$\text{Bulk Density} = r_{sol} \bullet (1 - \varepsilon_{bed}) \bullet (1 - \varepsilon_{particle})$$

In which:

r_{sol} =	solids density of the materials in the particle
$\varepsilon_{particle}$ =	particle porosity

[0028] Based on these equations, the particle porosity can be derived from the following experiments:

[0029] A glass tube with a diameter of 16.3 mm, containing a glass filter (pore diameter 40-90μm) as support for the powder, is filled with a known amount of powder (particle size between 355 and 710μm). The height of the powder bed is recorded. An airflow of 375 cm^3/min is flowed through the bed of powder. The pressure drop over the bed is measured. The pressure drop over the empty tube should also be measured at the specified air flow.

[0030] This measurement is repeated with the same quantity of powder, but now a more dense bed packing is achieved by gentle tapping of the tube containing the powder. Again the pressure drop Is measured at the specified air flow.

[0031] In order to be able to derive the particle porosity from these measurements, also the solids density of the particles is needed (eq. 2). This is measured using helium pycnometry, e.g. by using a penta pycnometer supplied by Quantachrome.

[0032] Based on the above described measurements and equations, the particle porosity can easily be derived.

[0033] For the purposes of the present invention, powder flow is defined in terms of the dynamic flow rate (DFR), in ml/s, measured by means of the following procedure. The apparatus used consists of a cylindrical glass tube having an internal diameter of 35 mm and a length of 600 mm. The tube is securely clamped in a position such that its longitudinal axis is vertical. Its lower end is terminated by means of a smooth 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.

[0034] To determine the DFR of a powder sample, the outlet orifice is temporarily closed, for example, by covering with a piece of card, and powder is poured through a funnel into the top of the cylinder until the powder level is about 10 cm higher than the upper sensor; a spacer between the funnel and the tube ensures that filling is uniform. The outlet is then opened and the time t (seconds) taken for the powder level to fall from the upper sensorto the lower sensor is measured electronically. The measurement is normally repeated two or three times and an average value taken. If V is the volume (ml) of the tube between the upper and lower sensors, the DFR (ml/s) is given by the following equation:

$$DFR = \frac{V}{t} \cdot ml/s$$

[0035] The averaging and calculation are carried out electronically and a direct read-out of the DFR value obtained.

[0036] The particulate starting material preferably comprises a builder, most preferably aluminosilicate, for example zeolite 4A or zeolite A24 or a salt, preferably an inorganic salt. Salts, preferably sodium, of phosphates, for example sodium tripolyphosphate (STP), carbonate, bicarbonate and sulphate are also suitable.

[0037] Other solid materials (ff required) may also be incorporated in the particulate starting material, although they may altematively or additionally be dosed at any appropriate stage(s) of the mechanical mixing.

[0038] The liquid component preferably contains at least one liquid nonionic surfactant. It may also contain one or more acid precursors of anionic surfactants and/or fatty acids. The acid precursor(s) can then be neutralised to form the corresponding anionicsurfactant(s) and the fatty acid(s) saponified by dosing one ormore suitable alkaline materials at an appropriate stage during the mechanical mixing process. Suitable alkaline materials include alkali metal carbonates, e.g. Na_2CO_3, and hydroxides, e.g. NaOH. Such alkaline materials may be dosed in solid form or as aqueous solutions. It is also possible to partially neutralise/saponify such precursors or fatty acids in the liquid component prior to the mechanical mixing step.

[0039] The detergent composition suitably comprise anionic detergent active. This may be incorporated as a pre-neutralised material, desirably as a component of the particulate starting material, or may be neutralised in situ. In the latter case the acid precursor of the active is preferably neutralised using a solid neutralising agent, for example carbonate, which is desirably a component of the particulate starting material.

[0040] The detergent active material present in the composition may be selected for anionic, cationic, ampholytic, zwitterionic or nonionic detergent active materials or mixtures thereof. Examples of suitable synthetic anionic detergent compounds are sodium and potassium (C_9-C_{20}) benzene sulphonates, particularly sodium linear secondary alkyl (C_{10}-C_{15}) benzene sulphonates (LAS); sodium or potassium alkyl sulphates (PAS); and sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum.

[0041] Suitable nonionics which may be employed include, in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amines or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Specific nonionic detergent compounds are alkyl (C_6-C_{22}) phenol ethylene oxide condensates, generally having 5 to 25 EO, i.e. 5 to 25 units of ethylene oxide per molecule, and the condensation products of aliphatic (C_8-C_{18}) primary or secondary linear or branched alcohols with ethylene oxide, generally 5 to 50 EO.

[0042] The level of detergent active material present in the composition may be in the range from 1 to 50% by weight depending on the desired applications. Nonionic material may be present in particulate starting material at a level which is less than 10% by weight more preferably less than 5% by weight and/or employed as the liquid binder in the mixing process optionally with another liquid component, for example water.

[0043] Suitably the particulate starting material constitutes 30 to 70% of the detergent composition.

[0044] Optionally, a layering material may be employed during the mixing step to control granule formation and reduce or prevent over-agglomeration. Suitable materials include aluminosilicates, for example zeolite 4A. The layering material is suitably present at a level of 1 to 4 wt %.

[0045] The composition may be used as a complete composition in its own right or may be mixed with other components or mixtures and thus may form a major or minor part of a final product. The composition may be blended with for example a spray-dried base powder. Conventional additional components such as enzymes, bleach and perfume may also be admixed with the composition as desired to produce a fully formulated product.

[0046] The present invention will now be further illustrated by the following non-limiting Examples.

EXAMPLES

[0047] All Examples used the following equipment: a Fukae FS30 for batch NTR experiments.

[0048] Unless stated otherwise herein, all amounts expressed as percentages are on a weight basis and based on the totai weight of the detergent composition or component prior to the addition of any post-dosed ingredients.

Production of zeolite-NTR powders according to the invention

[0049] The following slurries were spray dried to produce powders of high porosity and low bulk density (BD):

	Slurry 1 (wt%)	Slurry 2 (wt%)
Zeolite A24	40.0	43.8
LAS	0.0	1.3
Sokalan CP5	10.0	5.0
water	50.0	49.9

[0050] The resulting powders had the following properties:

Properties	Base Powder 1	Base Powder 2
BD, [g/l]	629	370
DFR, [ml/s]	115	88
d_{50} [μm]	210	279
RRd [μm]	242	299
RRn [-]	2.7	2.4
Moisture Content [%]	5-7	7
Particle Porosity	0.51	0.70
RRd = Rosin Rammler diameter RRn = Rosin Rammler number		

[0051] Sokalan CP5 is a polyacrylate/polymaleate copolymer.

[0052] The spray-dried zeolite-based porous carriers were subsequently used as base powders in NTR processes as described in Examples 1 and 2.

Examples 1 & 2

[0053] Both base powders were used in a batch NTR trial on a Fukae.

Formulation	Example 1 (wt%)	Example 2 (wt%)	Reference (wt%)
Base Powder 1	43.4		
Base Powder 2		46.4	
Zeolite A24			46.4
PAS adjunct	31.9	33.8	33.9
Nonionic 7EO	9.4	10.0	10.0
Nonionic 3EO	6.3	6.6	6.6
Fatty acid	2.5	2.6	2.6
NaOH	0.6	0.7	0.7
Zeolite A24 layering	5.6	0.0	0.0
Premix			
Agitator rpm	200		
Chopper rpm	3000		
Time [sec]	10		
Granulation			
Agitator rpm	100		

(continued)

Granulation			
Chopper rpm Time [min] Layering [sec]	3000 1-0.5 10	1	1
Powder properties			
DFR [mVs] RRd [μm] RRn	140 492-574 2.6	90 366 1.9	55 1015 1.6

[0054] The PAS adjunct used in the trial had the following composition:

PAS	45 wt%
Zeolite	38 wt%
Carbonate	9 wt%
Water + other components	8 wt%

<u>Production of STP-NTR powders according to the invention</u>

[0055] The following slurries were spray dried to produce powders of high porosity and low BD:

Composition	Wt%
STP (Rhodiaphos H5)	38.8
LAS	1.1
50% NaOH soln	0.3
45% Alkaline silicate soln	12.0
Water	47.9

[0056] The resulting powder had the following properties:

Properties	
BD [g/l]	404
DFR [ml/s]	111
d_{50} [μm]	303
RRd [μm]	349
RRn [-]	2.9
Moisture content [%]	5.9
Particle porosity	0.67

[0057] The spray-dried STP-based carrier was used to formulate powders in Examples 3 and 4.

<u>Examples 3 & 4</u>

[0058] The STP-based carrier was used in a batch NTR process using a Fukae FS30 mixer as follows:

	Example 3 (kg)	Example 4 (kg)	Reference
Standard STP	0	0	4.7
Spray-dried STP carrier	4.7	4.7	0
Sodium carbonate	5.2	5.2	5.2

(continued)

	Example 3 (kg)	Example 4 (kg)	Reference
Zeolite 4A (Wessalith P)	1.0	1.0	1.0
LAS acid	3.3	3,3	3.3
Zeolite 4A layering	0	0.3	0
Pre-mixing			
Pre-mix time (sec.)	10	10	10
RPM (agitator/chopper)	100/3000 Mixing	100/3000	100/3000
RPM (agitator)	100	200	100
RPM (chopper)	3000	3000	3000
Mixing time (sec)	120	120	120
Powder properties			
BD [g/l]	576	688	846
DFR [ml/s]	110	119	132
RRd [μm]	486	373	680
RRn [-]	1.72	1.70	1.19

[0059] Again the powders produced with porous carriers have a lower BD and a narrower particle size distribution as indicated by the higher RRn value.

Claims

1. A process for the production of a detergent powder composition having a bulk density of no more than 750 g/l, the process comprising mixing a particulate starting material which contains no more than 10% by weight of the starting material of detergent active material together with a liquid component comprising a detergent active material or a precursor therefor in a mixer/granulator having both a stirring and a cutting action **characterised in that** the starting material has a d_{50} average particle diameter of from 100μm to 1000μm and a particle porosity of at least 0.4 and that the stirrer is operated at a rate of 25 to 250 rpm and the cutter is operated at a rate of 300 to 3000 rpm.

2. A process according to claim 1 **characterised in that** the bulk density of the product detergent powder composition is controlled to a predetermined value by setting the operational speed of the mixer/granulator.

3. A process according to claim 1 or claim 2 **characterised in that** the starting material has a d_{50} average particle diameter of from 150μm to 800μm.

4. A process according to any preceding claim **characterised in that** the starting material has a d_{50} average particle diameter of from 200μm to 700μm.

5. A process according to any preceding claim **characterised in that** the starting material comprises a material formed by spray drying.

6. A process according to any preceding claim **characterised in that** the mixer/granulator is a high speed mixed densifier into which are dosed the starting material and the liquid component to form a granular material.

7. A process according to claim 6 **characterised in that** the material produced by mixing is subsequently dried and/or cooled.

8. A process according to any preceding claim **characterised in that** the mixer/granulator comprises a bowl-shaped vessel and a stirrer which rotates about a vertical stirrer axis.

9. A process according to any preceding claim **characterised in that** the particle porosity of the starting material is at least 0.45.

10. A process according to any preceding claim **characterised in that** the particle porosity of the starting material is at least 0.50.

Patentansprüche

1. Verfahren zur Herstellung einer Waschmittelpulverzusammensetzung mit einer Schüttdichte von nicht mehr als 750 g/l, wobei das Verfahren das Mischen eines partikelförmigen Ausgangsmaterials, das nicht mehr als 10 %, bezogen auf das Gewicht des Ausgangsmaterials, eines waschaktiven Materials enthält, mit einer flüssigen Komponente, die ein waschaktives Material oder eine Vorstufe dafür umfaßt, in einem Mischer/Granulator umfaßt, der sowohl eine Rühr- als auch eine Schnittwirkung hat, **dadurch gekennzeichnet, daß** das Ausgangsmaterial einen mittleren Partikeldurchmesser d_{50} von 100 μm bis 1000 μm und eine Porosität der Partikel von mindestens 0,4 hat, und dass der Rührer mit einer Geschwindigkeit von 25 bis 250 U/min und die Schnittvorrichtung mit einer Geschwindigkeit von 300 bis 3000 U/min betrieben wird.

2. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, daß** die Schüttdichte des Produktes der Waschmittelpulverzusammensetzung bei einem bestimmten Wert geregelt wird, indem die Arbeitsgeschwindigkeit des Mischers/Granulators eingestellt wird.

3. Verfahren nach Anspruch 1 oder Anspruch 2, **dadurch gekennzeichnet, daß** das Ausgangsmaterial einen mittleren Partikeldurchmesser d_{50} von 150 μm bis 800 μm hat.

4. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, daß** das Ausgangsmaterial einen mittleren Partikeldurchmesser d_{50} von 200 μm bis 700 μm hat.

5. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, daß** das Ausgangsmaterial ein durch Zerstäubungstrocknung erzeugtes Material umfaßt.

6. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, daß** der Mischer/Granulator ein Hochgeschwindigkeitsmischer/Verdichter ist, in den das Ausgangsmaterial und die flüssige Komponente dosiert werden, wodurch ein körniges Material erzeugt wird.

7. Verfahren nach Anspruch 6, **dadurch gekennzeichnet, daß** das durch Mischen hergestellte Material anschließend getrocknet und/oder abgekühlt wird.

8. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, daß** der Mischer/Granulator ein schalenförmiges Gefäß und einen Rührer, der um eine senkrechte Rührerachse rotiert, umfasst.

9. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, daß** die Porosität der Partikel des Ausgangsmaterials mindestens 0,45 beträgt.

10. Verfahren nach einem der vorstehenden Ansprüche, **dadurch gekennzeichnet, daß** die Porosität der Partikel des Ausgangsmaterials mindestens 0,50 beträgt.

Revendications

1. Procédé de production d'une composition de poudre détergente ayant une densité en masse ne dépassant pas 750 g/l, ledit procédé comprenant les étapes consistant à mélanger un matériau de départ en particules qui ne contient pas plus de 10 % en poids du matériau de départ de matériau détergent actif en même temps qu'un composant liquide comprenant un matériau détergent actif ou un précurseur de celui-ci, dans un mélangeur/granulateur produisant chacun une action de mélange et de découpe, **caractérisé en ce que** le matériau de départ a un diamètre moyen de particule d_{50} allant de 100 μm à 1000 μm et une porosité des particules d'au moins 0,4, et **en ce que** le mélangeur fonctionne à un rythme de 25 à 250 tours/minute et que la lame fonctionne à un rythme de 300 à 3000 tours/minute.

2. Procédé selon la revendication 1, **caractérisé en ce que** la densité en masse de la composition du produit détergent en poudre est contrôlée jusqu'à atteindre une valeur prédéterminée par le réglage de la vitesse de fonctionnement

du mélangeur/granulateur.

3. Procédé selon la revendication 1 ou la revendication 2, **caractérisé en ce que** le matériau de départ a un diamètre moyen de particule d_{50} allant de 150 μm à 800 μm.

4. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que** le matériau de départ a un diamètre moyen de particule d_{50} allant de 200 μm à 700 μm.

5. Procédé selon l'une quelconque des revendications précédentes **caractérisé en ce que** le matériau de départ comprend un matériau formé par séchage par vaporisation.

6. Procédé selon l'une des revendications précédentes, **caractérisé en ce que** le mélangeur/granulateur est un mélangeur/densificateur à grande vitesse dans lequel sont dosés le matériau de départ et le composant liquide afin de former un matériau granulaire.

7. Procédé selon la revendication 6, **caractérisé en ce que** le matériau produit par mélange est ensuite séché et/ou refroidi.

8. Procédé selon l'une quelconque des revendications précédentes **caractérisé en ce que** le mélangeur/granulateur comprend un récipient en forme de bol et un mélangeur qui tourne autour d'un axe mélangeur vertical.

9. Procédé selon l'une des revendications précédentes, **caractérisé en ce que** la porosité des particules du matériau de départ est d'au moins 0,45.

10. Procédé selon l'une quelconque des revendications précédentes, **caractérisé en ce que** la porosité des particules du matériau de départ est d'au moins 0,50.