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(71) Applicant: UNILEVER PLC
Unilever House Blackfriars P.O. Box 68
London EC4P 4BQ(GB)
(84) GB

Applicant: UNILEVER NV
Burgemeester s'Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam(NL)
(84) CH DE ES FR IT LI NL SE

(72) Inventor: Adams, Christopher John
'Highfield' 12 Fairview Road Oxton
Birkenhead Wirral Merseyside, L43 5SD(GB)
Inventor: Armstrong, Graeme Douglas
92 Thorn Drive
Little Sutton South Wirral, L66 4NJ(GB)
Inventor: Hight, Andrew Timothy
11A Venables Drive Spital
Bebington Wirral Merseyside, L63 9LY(GB)

(74) Representative: Stancliffe, Terence Christopher et al
UNILEVER PLC Patents Division P.O. Box 68
Unilever House
London EC4P 4BQ(GB)

(54) Enzyme-containing detergent compositions.

(57) A granular detergent composition or component therefor, which comprises:

(a) from 6% to 35%, often over 17%, by weight of non-soap detergent-active material consisting at least partially of anionic and/or nonionic detergent-active material;

(b) at least 45% by weight of builder

(b1) the builder including at least 28%, e.g. up to 45%, by weight (anhydrous basis) of crystalline or amorphous sodium aluminosilicate and/or citrate, carbonate, or layered silicate builder,

(c) a lipolytic enzyme the weight ratio of (b1) to (a) being from 0.9:1 to 2.6:1, preferably from 1.2:1 to 1.8:1; and other detergent ingredients to 100% by weight.

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ENZYME-CONTAINING DETERGENT COMPOSITIONS

This invention relates to detergent compositions, and in particular those containing lipolytic enzyme.

Detergent compositions comprising lipase are known. For example GB 1 372 034 (Unilever) discloses lipase from Pseudomonas in specific nonionic-containing [detergent] compositions for soaking fabrics.

USP 3 950 277 (Procter & Gamble) also describes fabric-soaking compositions: the described compositions comprise lipase and lipase activators and a number of lipases from microorganism and other sources are mentioned: those particularly mentioned as preferred are Amano CE, Amano M-AP, Takeda 1969-4-9, and Meito MY-30 lipases, but no indications are given of the form in which the lipase is to be prepared or used.

USP 4 011 169/NL 74 08763 (Procter & Gamble) describes the use of a similar range of enzymes in the preparation of additives for washing agents (detergent compositions). EP 0 214 761 (Novo) and EP 0 258 068 (Novo), each give detailed description of lipases from certain microorganisms, and also give certain uses in detergent additives and detergent compositions for the enzymes described. EP 0 214 761 gives detailed description of lipases derived from organisms of the species Pseudomonas cepacia, and certain uses therefor. EP 0 258 068 gives detailed description of lipases derived from organisms of the genus Thermomyces/Humicola, and certain uses therefor.

Also believed to be in use in certain areas is a lipase-containing granular detergent composition containing about 37% detergent actives including 5% nonionic detergent and the remainder substantially anionic detergent, about 16% zeolite, about 70-90 LU/g lipase, plus protease and other normal detergent additives.

An aim of this invention is the provision of a lipase-containing granular detergent composition with a particularly effective formulation for supporting the action of lipase enzyme.

According to the present invention, there is provided a granular detergent composition or component therefor, which comprises:

(a) from 6% to 35%, often over 17%, by weight of non-soap detergent-active material consisting at least partially of anionic and/or nonionic detergent-active material;

(b) at least 45% by weight of builder,

(b1) the builder including at least 28%, e.g. up to 45%, by weight (anhydrous basis) of crystalline or amorphous sodium aluminosilicate and/or citrate, carbonate, or layered silicate builder,

(c) a lipolytic enzyme, the weight ratio of (b1) to (a) being from 0.9:1 to 2.6:1, preferably from 1.2:1 to 1.8:1; and other detergent ingredients to 100% by weight.

According to the invention, certain of the compositions and components (adjuncts) therefor, including preferred compositions, can be prepared by treating a particulate starting material comprising:

(a) from 5 to 35% by weight of non-soap detergent-active material consisting at least partially of anionic detergent-active material, and

(b) from 28 to 45% by weight (anhydrous basis) of crystalline or amorphous sodium aluminosilicate, the weight ratio of (b) to (a) being at least 0.9:1, and other detergent components to 100% by weight, in a high-speed mixer in the presence of a liquid binder but in the absence of a finely divided particulate agent for improving surface properties, whereby granulation and densification to a bulk density of at least 650 g/litre are effected, and adding a lipolytic enzyme preparation after the granulation stage which has been carried out in the high-speed mixer.

Certain embodiments of the invention have a high bulk density, e.g. a bulk density of at least 650 g/litre.

It is useful and surprising that lipase activity is particularly well supported in the presence of the quantities of builder set out herein.

The lipolytic enzyme can usefully be added in the form of a granular composition of lipolytic enzyme with carrier material.

The added amount of lipolytic enzyme can be chosen within wide limits, for example 30 to 30,000 LU/g of granular detergent composition, e.g. at least 300 LU/g, often usefully at least 500 LU/g, sometimes preferably above 1000 LU/g or above 3000 LU/g or more.

Ihe lipolytic enzyme can be chosen from among a wide range of lipases: in particular the lipases described in for example the following patent specifications, EP 0214761 (Novo), EP 0258068 (Novo) and especially lipases showing immunological cross-reactivity with antisera raised against lipase from Thermomyces lanuginosus ATCC 22070, EP 0205208 (Unilever) and EP 0206390 (Unilever), and especially lipases showing immunological cross-reactivity with antisera raised against lipase with Chromobacter viscosum var lipolyticum NRRL B-3673, or against lipase from Alcaligenes PL-679, ATCC 31371 and FERM-P 3783, also the lipases described in specifications WO 87/00859 (Gist-Brocades) and EP 0204284

(Sapporo Breweries). Suitable in particular are for example the following commercially available lipase preparations: Novo Lipolase, Amano lipases CE, P, B, AP, M-AP, AML, and CES, and Meito lipases MY-30, OF, and PL, also esterase MM, Lipozym, SP225, SP285, Saiken lipase, Enzeco lipase, Toyo Jozo lipase and Diosynth lipase (Trade Marks).

It has now been surprisingly found that lipolytic enzyme exerts a comparatively greater effect when used in compositions of the type described above, than when used in comparative compositions as illustrated in the lipolytic example below.

According to preferred embodiments, the invention provides detergent powders combining high bulk density, good powder properties and excellent washing and cleaning performance, that can be prepared easily and conveniently.

These detergent compositions owe their combination of excellent properties and ready processability to a moderate content of surfactant, at least part of which is anionic, and a relatively high level of sodium aluminosilicate builder. It has been found that when the absolute amounts of aluminosilicate builder and surfactant, and the ratio of one to the other, are suitably chosen, the spray-dried base powder may be pulverised and granulated in a high-speed mixer without the need for the use of an "agent for improving surface properties" during the granulation step as prescribed by JP 61 069897A (Kao). The resulting dense granulate has good flow properties and is at least equal in washing and cleaning performance and cold water dispersability to the compositions described in the Kao specification which contain substantially higher levels of surfactant.

The aluminosilicate builder present in the compositions may be crystalline or amorphous or a mixture thereof, and can have the general formula

0.8-1.5 Na_2O . Al_2O_3 . 0.8-6 SiO_2.

These materials contain some bound water and should in most cases have a calcium ion exchange capacity of at least about 50 mg CaO/g. The preferred aluminosilicates contain 1.5-3.5 SiO_2 units (in the formula above) and have a particle size of not more than about 100 microns, preferably not more than about 20 microns. Both amorphous and crystalline aluminosilicates can be made readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

Crystalline aluminosilicates (zeolites) are preferred for use in the present invention. Suitable materials are described, for example, in GB 1 473 201 (Henkel) and GB 1 429 143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolites A and X, and mixtures thereof. Especially preferred for use in the present invention is Type 4A zeolite.

The ratio of aluminosilicate builder (anhydrous basis) to total non-soap surfactant in the compositions is preferably within the range of from 1.2:1 to 1.8:1.

The non-soap surfactant present consists at least partially of anionic surfactant. Suitable anionic surfactants are well known to those skilled in the art, and include linear alkylbenzene sulphonates, primary alcohol sulphates, alkyl ether sulphates, alpha-olefin sulphonates, internal olefin sulphonates, fatty acid ester sulphonates and combinations thereof.

If desired, nonionic surfactant may also be present, preferably in a minor amount. Suitably the surfactant component of the compositions of the invention may be constituted by 10 to 35% by weight of anionic surfactant and 0 to 10% by weight of nonionic surfactant.

If desired, soap may also be present, to provide foam control and additional detergency and builder power; soap is not included in the 17 to 35% figure given above for the total surfactant content of the compositions.

The compositions preferably do not contain more than 5% by weight of phosphate builders, and are more preferably substantially free of phosphate builders.

The particulate starting composition may be prepared by any suitable tower or non-tower method, for example, spray-drying or dry-mixing; spray-drying is preferred.

The final granulate may be used as a complete detergent composition in its own right. Alternatively, additional ingredients such as enzymes, bleach and perfume that are not suitable for undergoing the granulation process and the steps that precede it may be admixed to the granulate to make a final product.

In preferred cases the final granulate has a bulk density of at least 650 g/litre and more preferably at least 700 g/litre. It is preferably also characterised by an especially low particle porosity, not exceeding 25% and preferably not exceeding 20%, which distinguishes it from even the densest powders prepared by spray-drying alone.

In suitable examples of processes for making the preferred high-bulk-density compositions, a particulate starting material (detergent base powder) prepared by any suitable method is treated in a high-speed mixer to increase its bulk density and simultaneously to improve its powder properties. The process is especially useful for the densification of a spray-dried powder, and provides a route for the production of very dense granular detergent compositions, having excellent cleaning performance, containing low to moderate levels of anionic surfactant and high levels of aluminosilicate builder.

A preferred starting powder comprises:

(a) from 17 to 35% by weight of non-soap detergent-active material consisting at least partially of anionic detergent-active material, and

(b) from 28 to 45% by weight of crystalline or amorphous sodium aluminosilicate, the weight ratio of (b) to (a) being from 0.9:1 to 2.6:1, and other detergent components to 100% by weight.

On treatment in a high-speed mixer by an example of the process set out above, this can gives a granular detergent composition or component of the preferred high bulk density: it will be noted, however, that the process also gives good results with compositions containing lower levels of detergent-active material.

For carrying out the process, a mixer is apparently required that combines a high energy stirring input with a cutting action. The Fukae (Trade Mark) FS-G mixer manufactured by Fukae Powtech Kogyo Co., Japan, has been found to give excellent results in batchwise operation. This apparatus is essentially in the form of a vessel accessible via a top port, 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.

Other mixers suitable for use in the process include the Diosna (Trade Mark) V series ex Dierks & Söhne, Germany; the Lödige (Trade Mark) FM series ex Morton Machine Co. Ltd., Scotland; and the Pharma Matrix (Trade Mark) ex T K Fielder Ltd., England. Other mixers believed to be suitable for use in the process are the Fuji (Trade Mark) VG-C series ex Fuji Sangyo Co., Japan; the Lödige MTG ex Morton Machine Co. Ltd., Scotland; and the Roto (Trade Mark) ex Zanchetta & Co srl, Italy. The Lödige FM mixer differs from the Fukae mixer mentioned above in that its stirrer has a horizontal axis; this configuration is suitable for continuous operation.

As indicated above, the use of a high speed mixer is essential in the process set out above to effect granulation and densification. Some starting powders require pulverisation before granulation can take place; whether or not this is necessary depends, among other things, on the method of preparation of the starting powder and its free moisture content. Powders prepared by spray-drying, for example, are more likely to require pulverisation than powders prepared by dry-mixing. Pulverisation may, if desired, be carried out as a separate process step, in any suitable separate apparatus, before granulation in the high speed mixer is carried out. It is convenient, however, to use the same high-speed mixer for the pulverisation step as for the granulation step. The high speed mixer may be used for the pulverisation step by running the mixer at high speed, using both stirrer and cutter. A relatively short residence time (for example, 2-4 minutes for a 35 kg batch) is generally sufficient. A mass of finely divided powder is obtained.

The granulation step follows, and it is during that stage that densification to very high values of at least 650 g/litre, preferably at least 700 g/litre can be achieved, giving a dense, granular product of very uniform particle size and generally spherical particle shape. The final bulk density can be controlled by choice of residence time, and it has been found that the powder properties of the resulting granulate are best if the bulk density has been allowed to rise to at least 650 g/litre.

The presence of a liquid binder is necessary for successful granulation. The amount of binder added preferably does not exceed that needed to bring the free moisture content of the composition above about 6% by weight, since higher levels lead to a deterioration in the flow properties of the final granulate. If necessary, binder, preferably water, may be added before or during granulation, but some starting powders will inherently contain sufficient moisture. In the Fukae mixer, granulation is effected by running the mixer at a relatively high speed using both stirrer and cutter; a relatively short residence time (for example, 5-8 minutes for a 35 kg batch) is generally sufficient. If a liquid binder is to be added, it may be sprayed in while the mixer is running. In one preferred mode of operation, the mixer is first operated at a relatively slow speed while binder is added, before increasing the speed of the mixer to effect granulation.

If the starting powder has a sufficient free moisture content to render the addition of a binder unnecessary, pulverisation (if required) and granulation need not be regarded as separate process steps but as one single operation. Indeed, it is not, in that case, necessary to decide in advance whether or not pulverisation is required: the mixer may simply be allowed to do what is necessary, since the mixer conditions required are substantially the same for pulverisation and for granulation.

In general, it is highly preferred that during granulation no "agent for improving surface properties" as defined in the above-mentioned JP 61 069897A (Kao) be present. When processing a formulation having a relatively high ratio of aluminosilicate builder to surfactant, in accordance with examples of the present invention, the use of a finely divided particulate material such as fine sodium aluminosilicate during the granulation step is not only unnecessary but can with some formulations make granulation more difficult, or even impossible.

In accordance with a preferred embodiment of the invention, a finely divided particulate flow aid may be admixed with the granular material after granulation is complete. Advantageously, flow aid is added while the granulate is still in the high-speed mixer, and the mixer is operated at a slow speed for a further short period. No further granulation occurs at this stage. It is also within the scope of the invention to add the flow aid to the granulate after removing the latter to different apparatus.

This embodiment of the invention should be distinguished from the prior art process of JP 61 069897A (Kao), mentioned above, in which an "agent for improving surface properties", which can be fine sodium aluminosilicate, is present during the granulation stage itself. It is within the scope of the present invention to add a particulate flow aid after granulation is complete, but, as explained above, it is essential to the invention that no finely divided particulate "agent for improving surface properties" be present during granulation. The addition of a flow aid after granulation is complete can have an additional beneficial effect on the properties of the granulate, regardless of the formulation, whereas the presence of this type of material during the granulation step in the present process makes processing more difficult.

According to a preferred embodiment of the process, granulation is carried out at a controlled temperature somewhat above ambient, preferably above 30 deg C. The optimum temperature is apparently formulation-dependent, but appears generally to lie within the range of from 30 to 45 deg C, preferably about 35 deg C. This temperature may also be maintained during the subsequent admixture of a flow aid.

The flow aid is a finely divided particulate material. The preferred average particle size is 0.1 to 20 microns, more preferably 1 to 10 microns. Substantially amorphous materials are preferred.

According to a highly preferred embodiment, the flow aid is finely divided amorphous sodium aluminosilicate. This material is effective in improving flow properties even at very low levels, and also has the effect of increasing bulk density. It is therefore possible to adjust bulk density by appropriate choice of the level of amorphous sodium aluminosilicate added after granulation.

A suitable material is available commercially from Crosfield Chemicals Ltd, Warrington, Cheshire, under the trade mark Alusil. Other flow aids manufactured by this company include Neosyl (Trade Mark) precipitated silica and Microcal (Trade Mark) precipitated calcium silicate.

The flow aid is advantageously used in an amount of from 0.2 to 5.0% by weight, based on the starting powder, more preferably from 0.5 to 3.0% by weight.

Zeolite (hydrated crystalline sodium aluminosilicate) is substantially less effective as a flow aid in the context of the present invention. Substantially higher levels than those quoted above are needed before any flow or bulk density benefit is observed.

The invention is illustrated by the following non-limiting Examples, in which parts and percentages are by weight unless otherwise stated.

Example 1

A detergent composition is prepared to the following composition by spray-drying an aqueous slurry to a free moisture content of substantially zero:

	parts
Linear alkylbenzene sulphonate	24.0
Nonionic surfactant	2.0
Soap	1.0
Zeolite (anhydr.)	38.0
Water bound with zeolite	10.84
Sodium silicate	4.0
Acrylate/maleate copolymer	2.0
Minor ingredients	2.0
Sodium carbonate	10.0
	94.64

The ratio of zeolite (anhydrous) to non-soap surfactant in this composition is 1.46.

35 kg of this spray-dried powder are introduced into a Fukae (Trade Mark) FS-G series high speed mixer, and pulverised at high speed for 2-4 minutes. Water (2.0 parts) is then sprayed in while the mixer is

allowed to run at a slower speed, then the speed is increased for 5-8 minutes while maintaining the temperature at about 35° C. During this period granulation occurs.

A sample of the granular product is removed from the mixer. It is generally free-flowing and shows no tendency to cake. Its dynamic flow rate can for example be about 65 ml/s.

1.0 part of Alusil (Trade Mark) fine amorphous sodium aluminosilicate is introduced into the Fukae mixer, which is then operated at a slow speed for 1 minute. The resulting granular product is free-flowing and generally shows no tendency to cake. In representative runs, its bulk density can be about 740 g/litre, its mean particle size about 405 microns and its dynamic flow rate about 105 ml/s.

The following ingredients are then mixed with the granular material to give 99 parts of final detergent powder:

Coloured speckles	1.5 parts
Alcalase (TM))	0.61 parts
Lipase to required extent eg	0.5 parts
Perfume	0.25 parts

(Lipase enzyme is postdosed to the required extent after the main body of preparative steps described).

Examples 2 and 3

35 kg of the spray-dried powder used in Example 1 are introduced into a Lödige (Trade Mark) FM series high speed mixer, and pulverised for 4 minutes. Water (1.1 kg, 3.5%) is then sprayed in while the mixer continues to run at the same speed, then the mixer is allowed to run for a further 3 minutes while the temperature was maintained at about 35° C. During this period granulation occurs. A sample (Example 2) is removed from the mixer and its powder properties determined: these are for examples as shown in Table 1 below.

Alusil (Trade Mark) finely divided amorphous sodium aluminosilicate (1.2 kg) is then introduced into the mixer which is allowed to run for a further 0.5 minutes. Physical properties of an example of the resulting powder (Example 3) are shown in Table 1 below, from which the beneficial effect on flow and bulk density of adding a flow aid after granulation is complete is apparent. The presence of the Alusil did result in an increase in the content of fine particles < 180 microns, but not to an unacceptable level.

Table 1

Example	2	3
Bulk density (g/l)	680	754
Dynamic flow rate (ml/s)	100	109
Particle size (microns)	573	524
Fines content (wt% of particles < 180 microns)	0	15

Examples 4 to 6

Three powders are prepared by spray-drying the nominal composition shown in Example 1 to three different moisture contents, as shown in Table 2 below. Since the 38.0 parts of zeolite (anhydrous basis) in the formulation require 10.84 parts of water of hydration, the free moisture content of each powder is found by subtracting that figure from the total moisture content: it will be noted that the powder of Example 4 is overdried, while that of Example 6 contains 3.16 parts of free moisture.

A 10 kg batch of each powder is granulated (and, where necessary, pulverised) in a Diosna (Trade Mark) V series mixer, using an agitator speed of 196 r p m and a chopper speed of 3000 r p m. In the case of Examples 4 and 5, where pulverisation initially occurred, water is sprayed into the mixer, in the amount and for the time indicated, before granulating. After granulation is complete, Alusil finely divided amorphous sodium aluminosilicate (0.1 kg) is admixed to the granulate. Lipase enzyme is postdosed to the required extent (e.g. 0.5%) after the main body of preparative steps described. Properties of examples of the three granulates are shown in Table 2.

Table 2

Example	4	5	6
Total moisture			
(wt %)	8.0	11.0	14.0
Pulverisation			
time (min)	4	3	-
Water addition:			
amount (kg) time (min)	0.2 2	0.1 1	- -
Granulation			
time (min)	4	3	4
Bulk density			
(g/l)	740	832	735
Dynamic flow			
rate (ml/s)	114	120	120
Compressibility			
(% v/v)	8.8	9.4	8.8
Particle size			
(microns) Fines content (wt% < 180 microns)	483 5.0	553 2.4	521 6.1

Example 7

The effectiveness of lipolytic enzyme in a detergent composition of the type described above, and the comparative effectiveness of lipolytic enzyme in a comparative detergent formulation, was estimated as follows.

(a) Triolein-soiled cotton and polyester materials were washed in a pH-stat under uniform conditions using two detergent formulations as given below.

(b) pH-stat titration of the acidity developed by free fatty acid (FFA) during the wash was carried out to show an index quantity of free fatty acid per standard test cloth indicating the relative effectiveness of lipolytic action.

The detergent formulations were as follows:

	(a)	(b)
	%	%
linear alkylbenzene sulphonate	20	26
coco-PAS	-	8
nonionic detergent	2	4
soap	1	3
zeolite (anhydrous)	35	18
polymer	1.5	1
sodium carbonate	10	20
sodium sulphate	11	4
sodium silicate	4	6
sodium carboxymethylcellulose	0.75	-
sodium succinate	2	-
ratio b1:a	1.59	0.47

Lipase was used in each case in an amount corresponding to 5 LU/ml wash solution, and the wash water had 4 degrees FH Calcium, 2 degrees FH magnesium. Wash temperature was 30 degrees C. Cloth:liquor ratio was 1:80, the cotton was soiled with 6.4% by weight triolein and the polyester had 10% by weight triolein.

After washing with formulation (a) the test showed the presence of 2.3 micromoles of FFA produced in the wash liquor per cotton test cloth and 7.6 micromoles of FFA per polyester test cloth.

After washing with comparative formulation (b) the test showed the presence of 1.0 micromoles of FFA produced in the wash liquor per cotton test cloth and 6.1 micromoles of FFA per polyester test cloth.

It was apparent that formulation (a) enabled comparatively more lipolytic activity from the given quantity of lipolytic enzyme.

Example 8

A preferred detergent composition was prepared substantially according to the method of Example 1 but with the following composition:

	% by weight
Linear alkylbenzene sulphonate	25
Nonionic surfactant (7EO)	2
Soap (fully hardened tallow soap)	1
Zeolite (anhydr.)	36
Water bound with zeolite	10
Alkaline sodium silicate	4
Acrylate/maleate copolymer CP5	1
Sodium carbonate	16
Sodium sulphate	2
Na caroxymethylcellulose	1
Enzymes	0.6
Minors and water	to 100

The lipase (preferably Lipolase) is added as described above, and is preferably present in addition to protease (Alcalase).

The invention is susceptible of modifications and variations within the scope of the preceding disclosure with Examples, and extends to all combinations and subcombinations of the features mentioned or incorporated.

Claims

1. A granular detergent composition or component therefor, which comprises:
(a) from 6% to 35% by weight of non-soap detergent-active material consisting at least partially of anionic and/or nonionic detergent-active material;
(b) at least 45% by weight of builder,
(b1) the builder including at least 28% by weight (anhydrous basis) of crystalline or amorphous sodium aluminosilicate and/or citrate, carbonate, or layered silicate builder,
(c) a lipolytic enzyme,
the weight ratio of (b1) to (a) being from 0.9:1 to 2.6:1, preferably from 1.2:1 to 1.8:1; and other detergent ingredients to 100% by weight.

2. A composition as claimed in claim 1, which has been prepared by treating a particulate starting material comprising: (a) from 5 to 35% by weight of non-soap detergent-active material consisting at least partially of anionic detergent-active material, and (b) from 28 to 45% by weight (Anhydrous basis) of crystalline or amorphous sodium aluminosilicate, the weight ratio of (b) to (a) being at least 1.9:1, and other detergent components to 100% by weight in a high-speed mixer in the presence of a liquid binder but in the absence of a finely dividied particulate agent for improving surface properties, whereby granulation and densification to a bulk density of at least 650 g/litre are effected, and adding a lipolytic enzyme preparation after the granulation stage which has been carried out in the high-speed mixer.

3. A composition as claimed in claim 1 or 2, which has a bulk density of at least 650 g/litre.

4. A composition as claimed in claim 1, 2 or 3, in which the lipolytic enzyme is Lipolase (TM).

5. A composition as claimed in any of claims 1-4, in which the lipolytic enzyme is present at from about 300-3000 LU/g.

6. A composition as claimed in any of claims 1-5, in which the proportion of non-soap detergent-active material is over 17% by weight.

7. A composition as claimed in any of claims 1-6, in which the builder includes 28-45% by weight of component (b1).

8. A detergent composition substantially as described in relation to any one of the foregoing Examples.