(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 550 086 A1**

(12) EUROPEAN PATENT APPLICATION

(21) Application number: **92203804.7**

(22) Date of filing: **08.12.92**

(51) Int. Cl.^{5}: **C11D 11/02**, C11D 3/10, C11D 3/12, C11D 1/12

(30) Priority: **31.12.91 US 816366**

(43) Date of publication of application:
07.07.93 Bulletin 93/27

(84) Designated Contracting States:
CH DE ES FR GB IT LI NL SE

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(54) Process for making a granular detergent composition.

(57) A process is provided for the manufacture of a granular detergent composition comprising the steps of:
(i) mixing a carrier component comprising from 5% to 95% by weight of a non-soap surfactant, with a solid component to form a sprayable slurry; and
(ii) solidifying the slurry into granules by spray-cooling.

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(3.10/3.6/3.3.1)

Technical Field

The present invention relates to a process for making a granular detergent composition.

Background and Prior Art

In the main, commercial granular detergents are produced by spray-drying. In spray-drying the detergent components, such as surfactants and builders are mixed with as much as 35 to 50% by weight of water to form a slurry, heated and then spray-dried.
The process is expensive since the energy requirement for heating and spray-drying the slurry is large.

Granular detergents produced by spray-drying have good solubility but tend to be of low bulk density and therefore, have large packing volumes. High bulk density granular detergent compositions have several advantages over low bulk density compositions. The packing volume of the granules is lower, meaning that packaging can be smaller which facilitates storage and transport of products.

Since spray-drying is a hot process it also places restrictions on what can be included in the formulation, in particular nonionic surfactants which can give rise to unwanted emissions and sensitive or volatile components such as enzymes and perfumes which could be degraded or lost.

There are many prior art non spray-drying processes which produce granular detergent. These have drawbacks as well. Most, in a first stage, form a dough which is de-agglomerated in a second stage to form granules. Usually the granules are then coated and dried in further stages. Such multi-stage processes require more than one mixer and a separate granulation operation. Other processes require use of the acid form of the surfactant to work.

Some others require high temperatures which degrade the starting materials. For the manufacturer these non-spray-drying routes to granular detergents require substantial investment in machinery.

In the past it has been proposed that non-automatic washing machine powders be produced by a process of spray-cooling.

U.S. Patent 4,466,897 (Appel) discloses a process for making a detergent powder for use in a non-automatic washing machine by impregnating spray-dried or spray-cooled washing powder comprising a sodium soap as the principal organic detergent-active species, with a finely divided, water soluble sodium salt.

British Patent Specification 1,344,253 (Herlow) discloses a process for producing an enzymatic adjunct for use in detergent compositions by suspending enzyme particles in a molten nonionic surfactant and solidifying the mixture by spray-cooling. The production of detergent compositions comprising anionic surfactants is not disclosed.

There is a need for a process to make a granular detergent composition, which mitigates the disadvantages of the prior art processes and allows granular detergent compositions with non-soap, anionic surfactants to be produced.

We have now found that it is possible to make granular detergent compositions in a simple process, using readily available equipment and starting materials. Using this same process, granular detergent compositions of high bulk density can be produced that is of a bulk density greater than 600 g/l.

Definition of the invention

The invention provides a process for the manufacture of a granular detergent composition comprising the steps of

(i) mixing a carrier component comprising from 5% to 95% by weight of non-soap surfactant with a solid component to form a sprayable slurry; and

(ii) solidifying the slurry into granules by spray-cooling.

The advantages of this process are that it uses existing equipment since most granular detergents are produced by spray-drying and this same apparatus can be used for spray-cooling, operating costs are low since no drying gas is used in the process and nonionic surfactants can be used in the compositions without undesirable emissions. The process also gives the advantage of bulk density flexibility since high or low bulk density granules can be produced.

Detailed Description of the Invention

In the process a solid component, which can comprise detergency builders such as water-soluble alkaline inorganic materials (for example sodium carbonate seeded with calcium carbonate) , zeolite, sodium tripolyphosphate, other water-soluble inorganic materials, for example, sodium bicarbonate or silicate, fluorescers, polycarboxylate polymers, anti-redeposition agents, and fillers, is mixed with a carrier component which, in addition to a non-soap anionic surfactant can comprise water, silicate solution, liquid polymer components, polyethylene glycols, perfumes, enzymes and other materials.

The process is very flexible with respect to the chemical composition of the starting materials. Phosphate as well as zeolite based compositions and compositions having low or high surfactant contents may be made. The process is also suitable for preparing calcite/carbonate containing compositions.

The solid component is preferably particulate with particle size of 1 to 400 microns (as measured by Rosin Rammler), more preferably 1 to 350 microns, most preferably 10 to 300 microns. The solid component preferably comprises from 5 to 95% by weight of detergent builders, more preferably from 10 to 80%, most preferably from 20 to 60%.

The carrier preferably comprises from 5% to 95% by weight of non-soap surfactants, more preferably from 15% to 80% by weight, most preferably from 20% to 70% by weight.

Preferably, the carrier component comprises a mixture of surfactants for example, anionic, nonionic or zwitterionic.

The anionic surfactant may be selected from linear alkyl benzene sulphate or sulphonate preferably C_{12} to C_{18} linear alkyl benzene sulphate, alpha-olefin sulphate or sulphonate, internal olefin sulphate or sulphonate, fatty acid ester sulphate or sulphonate and primary and secondary alcohol sulphates or sulphonates.

In the context of the present invention by non-soap anionic surfactant is meant any anionic surfactant except the water-soluble salts of C_8 to C_{24} fatty acids.

The nonionic surfactant may be selected from those compounds produced by the condensation of alkylene oxide groups with an organic hydrophobic compound for example alkyl phenols or alcohols. Preferred nonionics are the water soluble condensation products of aliphatic alcohols with 8 to 30 carbon atoms in the molecule with 3 to 15 moles of ethylene oxide per mole of alcohol. Other nonionics include water soluble amine oxides containing one alkyl moiety of about 10 to 18 carbon atoms and 2 moieties selected from the groups of alkyl and hydroxyalkyl moieties with from 1 to 3 carbon atoms and those nonionics derived from sugars such as alkyl poly glycoside.

The zwitterionic surfactants include derivatives of aliphatic quaternary ammonium phosphonium and sulphonium compounds in which one of the aliphatic substituents contains an anionic water-solubilizing group.

Preferably, the carrier comprises a mixture of surfactants for example a mixture of linear alkylbenzene sulphonate containing from 11 to 14 carbon atoms and a C_{12} to C_{15} primary alcohol ethoxylated with 3 to 7 moles of ethylene oxide per mole of alcohol in a weight ratio of anionic to nonionic of 3 to 1 or a mixture of a C_{14} to C_{17} secondary alcohol sulphate with a $C_{12\ \mathrm{to}\ \mathrm{c}}15$ primary alcohol ethoxylated with 7 moles of ethylene oxide per mole of alcohol in a weight ratio of from 2 to 1.

Preferably, the carrier component is prepared by neutralizing a non-soap sulphonic acid with a mixture of nonionic surfactant and concentrated aqueous alkali metal hydroxide in an amount stoichiometric to the sulphonic acid. The sulphonic acid may also be under or over neutralized. The heat generated by the exothermic neutralization allows the carrier to be mixed with the solid component and pumped directly to the spray-cooling apparatus without further heating.

Preferably the surfactant comprises less than 20% by weight of water, more preferably less than 10% and most preferably less than 5% by weight and is made by the method described in US-A-4,923,636 (Blackburn) or US-A-4,826,632 (Blackburn) incorporated herein by reference. Preferred surfactant mixtures contain 20 to 80% by weight of anionic surfactant and 20 to 80% by weight of nonionic surfactant and no water.

Preferably the carrier component comprises less than 30% by weight of water, more preferably between 25% and 0%, most preferably between 10% and 0%.

The carrier component preferably comprises from 1% to 20% of a solidification aid such as a fatty acid (which can be neutralized to form a salt in situ) or a polymer with a melting point between 40 °C and 85 °C (for example a polyethylene glycol or polypropylene glycol of molecular weight between 1400 and 20,000.) Preferred solidification aids are polyethylene glycol of molecular weight 3350 and stearic acid. The carrier component preferably comprises from 2 to 10% by weight of a solidification aid.

The spray-cooling apparatus used to carry out the process consists essentially of a slurry mixer, pump and spray nozzle inside a cylindrical vessel fed with counter current air.

In the process according to the invention, the carrier may be prepared by any suitable method which maintains the total water content below 30% by weight of the carrier. It is preferred to mix any nonionic surfactant with concentrated aqueous sodium hydroxide solution in an amount stoichiometric to the anionic sulphonic acid, mix with the sulphonic acid and add a solidification aid such as fatty acid.

The carrier thus obtained is mixed with the solid component to form a slurry. The slurry is then atomized into droplets and solidified into granules either by reduction of temperature below the melting point of the slurry or by neutralization of any fatty acid present by coating the droplets with soda ash. Optionally the solidified droplets can be coated with a flow aid such as zeolite to improve their powder properties. The resulting granules are preferably from 300 to 1200 microns in diameter and are generally spherical with little or no internal porosity, especially if atomized without the aid of air injection. This has the effect that those granules with bulk density above 600 g/l produced according to the invention and without air injection comprise largely homogeneous interiors in which the voids are filled and internal porosity is low. Most prior art high bulk density granules comprise solids re-agglomerated into granules which have a larger internal porosity than the present spray cooled granule since voids between re-agglomerated solids are not filled. Prior art spray cooled soap powders consist of particles with large amounts of internal porosity since voids are created by spray cooling the superheated slurry.

The ratio by weight of carrier to solid component in the slurry is preferably from 1:2 to 4:1 by weight, more preferably 1:1 to 2:1.

The slurry temperature is preferably from 50 °C to 100 °C.

The invention is further illustrated by the following non-limiting examples, in which parts and percentages are by weight.

The following abbreviations are used:

LAS : Sodium salt of C_{11}-C_{15} alkylbenzene sulphonate acid (stephan Bio-Soft S-100)
PEG 3350 : Polyethylene glycol (mw 3350) Carbowax PEG 3350 ex Union Carbide
Zeolite : Zeolite 4A PQ Corp., Valfor 100
DSA : dense soda ash, FMC, Grade 260
NII3EO : nonionic surfactant (ethoxylated C_{12}-C_{15} alcohol) 13 moles of ethylene oxide per mole of alcohol.

Examples 1-2

Alkylbenzene sulphonic acid was neutralized with a mixture of nonionic surfactant and a stoichiometric amount of concentrated aqueous sodium hydroxide. Polyethylene glycol of molecular weight 3350 and zeolite were added to this mixture. Due to the neutralization reaction the slurry temperature was raised to about 800 °C. The slurry was then pumped through a gear pump to a two fluid nozzle isolated in an Aeromatic fluid bed model STREA-1 and atomized into droplets. Dense soda ash and zeolite were fluidized in the bed to coat the droplets which solidified in the bed.

Example 3

A slurry was produced as in Examples 1 and 2 by mixing in a spray pot. The slurry was then atomized into droplets using air pressure in place of the pump in Examples 1 and 2. It can be seen that this reduces the bulk density from the 845 and 852 g/l of Examples 1 and 2 to 750 g/l in Example 3.

Example 4-5

A slurry was prepared as in Example 3 and atomized using air pressure as in Example 3. The slurry was atomized into a cooling tower, the droplets landing on a bed of zeolite.

Example 6

A slurry was prepared as in Examples 1 and 2 and sprayed via a single fluid nozzle into a cooling tower. Light soda ash was blown into the bottom of the tower to form a cloud of soda ash which coated the falling particles.

Carrier	1	2	3	4	5	6
LAS	7.4	6.4	3.6	-	-	16.1
Coco PAS	-	-	-	18.7	12.5	-
NI13EO	7.4	6.4	3.8	-	-	15.1
NI11EO	-	-	-	-	-	-
NI7EO	-	-	-	3.4	2.2	-
NI3EO	-	-	-	21.6	14.4	-
Water	1.2	1.1	1.8	4.3	2.9	2.7
PEG 3350	4.9	4.2	-	-	-	-
Stearic Acid	-	-	0.6	-	-	1.5

Solids to Slurry	1	2	3	4	5	6
Zeolite	8.5	11.4	11.4	16	24	44.2
Dense Soda Ash	-	-	-	16	24	-
Sodium Citrate	-	-	-	-	-	-

Coating/Post Dose	1	2	3	4	5	6
Light Soda Ash	-	-	79	-	-	20
Dense Soda Ash	35.3	35.3	-	-	-	-
Zeolite	35.3	35.3	-	20	20	-

Slurry and Spraying Parameter	1	2	3	4	5	6
Slurry Temp (°C)	80	80	90	90	90	95
Viscosity (cP)	-	-	-	-	-	1700
Shear rate (/s)	-	-	-	-	-	250
Slurry Pressure (pasig)	35	20	80	90	90	200-800
Air Pressure (psig)	8	8	25	30	30	0

Solid Powder	1	2	3	4	5	6
Bulk Density	852	845	710	-	650	560
Flow Properties	good	good	good	good	good	fair

Claims

1. Process for the manufacture of a granular detergent composition comprising the steps of:

(i) mixing a carrier component comprising from 5% to 95% by weight of a non-soap surfactant, with a solid component to form a sprayable slurry; and

(ii) solidifying the slurry into granules by spray-cooling.

2. Process as claimed in claim 1, wherein the carrier component comprises less than 30% by weight of water.

3. Process as claimed in claim 1 or 2, wherein the non-soap surfactant comprises from 20% to 80% by weight of anionic and from 20% to 80% by weight of nonionic surfactant.

4. Process as claimed in any of claims 1-3, wherein the solid component comprises from 5 to 95% by weight of detergent builders.

5. Process as claimed in any of claims 1-4, wherein the solid component is particulate with particle sizes between 1 and 400 microns.

6. Process as claimed in any of claims 1-5, wherein the carrier component comprises from 1 to 20% by weight of a solidification aid.

7. Process as claimed in any of claims 1-6, wherein the slurry temperature is from 50°C to LOO°C prior to spraying.

8. Process as claimed in any of claims 1-7, wherein the resulting granules are from 300 to 1200 microns in diameter.

9. Process as claimed in any of claims 1-8, wherein the bulk density of the resulting granules is greater than 600 g/l.

10. A granular detergent composition produced by the process according to any of claims 1-9, said granular detergent composition consisting essentially of granules with low internal porosity.

11. Process for the manufacture of a granular detergent composition comprising the steps of:
(i) selecting a carrier component comprising a non-soap anionic surfactant;
(ii) selecting a solid component;
(iii)mixing the carrier and solid components to form a sprayable slurry;
(iv) solidifying the slurry into granules by spray cooling;
(V) coating the granules with a flow aid.

12. Process as claimed in claim 11 wherein the slurry is spray-cooled using air pressure.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 92203804.7

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
D,X	GB - A - 1 344 253 (GRINDSTEDVERKET) * Examples; claims 1-5 * --	1,2,5, 7,8	C 11 D 11/02 C 11 D 3/10 C 11 D 3/12 C 11 D 1/12
D,A	US - A - 4 466 897 (APPEL et al.) * Totality * ----	1-12	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

C 11 D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
VIENNA	10-03-1993	SEIRAFI

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
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E : earlier patent document, but published on, or after the filing date
D : document cited in the application
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& : member of the same patent family, corresponding document