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(54) Detergent compositions.

(57) A tablet of compacted detergent powder having a surfactant system comprising a nonionic surfactant, at least one detergency builder and optionally other detergent ingredients, is characterised in that the nonionic surfactant comprises a short-chain material having an average alkyl chain length of less than C_{12}. The benefit is improved disintegration and dissolution in the wash liquor.

EP 0 598 586 A1

TECHNICAL FIELD

The present invention relates to detergent compositions in tablet form containing nonionic surfactants.

BACKGROUND AND PRIOR ART

Detergent compositions in tablet form have been known for many years although the form has never achieved great popularity in the market. Tablets have various advantages over powdered products: for example, they do not require measuring and so are easier to dose, and they can be more economically stored as they are compact.

Detergent tablets are generally made by compressing or compacting a detergent powder. It has proved difficult to strike a balance between tablet strength and the ability to disintegrate and disperse in the wash liquor. Tablets formed using only a light compaction pressure tend to crumble and break up on handling and packing; while more strongly compacted tablets may be sufficiently cohesive but will then fail to disperse at a sufficient rate and to an adequate extent in the wash liquor.

Nonionic surfactants have beneficial cleaning characteristics when included in detergent formulations, especially in removing oily soil from fabrics. However, when in contact with aqueous solutions nonionic surfactants tend to form viscous phases which impede dissolution. In tablets the problem is exacerbated.

The problem with poor dissolution and dispersability has proved especially acute with tablets formed by compressing powders containing water-insoluble builders such as alkali metal aluminosilicates.

Detergent tablets containing nonionic surfactants have been disclosed in the art.

EP 355 626A (Henkel) discloses a detergent tablet containing zeolites, builder salts, anionic surfactants and nonionic surfactants. The tablet is made by mixing two preformed components (A) and (B), wherein (A) contains the whole amount of anionic surfactant and (B) contains 75-100 wt% of the nonionic surfactant. The tablets are claimed to have good resistance to breaking up prior to use yet dissolve quickly in the washing machine.

A detergent tablet containing a detergency builder and anionic detergent active has been disclosed in EP 466 485A (Unilever). The problem of poor dissolution is substantially alleviated by ensuring that the anionic detergent-active compounds present are not distributed widely through the tablet, but are concentrated in discrete domains within a continuous phase containing little or no anionic detergent-active compound. Nonionic surfactants may optionally be treated similarly.

It has now been discovered that the difficulty of producing robust tablets containing nonionic surfactants which dissolve and disintegrate quickly in the wash, without the need for any processing modifications or special segregation procedures, can be alleviated by the use of nonionic surfactants of strictly controlled alkyl chain length. This measure can also provide detergency benefits.

Particulate detergent compositions containing short-chain nonionic surfactants are disclosed in GB 1 460 646, GB 1 462 133, GB 1 462 134, GB 1 485 316 and GB 1 566 326 (Procter & Gamble); GB 1 519 433 and FR 2 303 850A (Rhone-Poulenc); EP 200 953A and WO 91 10718A (Henkel). However, the use of short-chain nonionic surfactants in detergent tablets to improve their disintegration and dissolution characteristics is not disclosed.

DEFINITION OF THE INVENTION

The present invention accordingly provides a tablet of compacted detergent powder comprising a surfactant system comprising a nonionic surfactant, at least one detergency builder and optionally other detergent ingredients, characterised in that the nonionic surfactant comprises a condensation product of ethylene oxide with an aliphatic alcohol having an average alkyl chain length of less than C_{12}.

The invention further provides the use of a nonionic surfactant which is a condensation product of ethylene oxide with an aliphatic alcohol having an average alkyl chain length of less than C_{12} to improve the disintegration and dissolution in the wash liquor of a tablet of compacted detergent powder.

DETAILED DESCRIPTION OF THE INVENTION

The Nonionic Surfactant

The detergent tablet of the invention is characterised in that its surfactant system comprises ethoxylated nonionic surfactant having an average alkyl chain length of less than C_{12}. This component will be referred to hereinafter as the short-chain nonionic surfactant.

Commercial nonionic surfactants are generally mixtures containing a spread of chain lengths around an average value. Preferably the surfactant system is free of nonionic surfactants which are commercial materials having an average chain length of C_{12} and above.

Preferably the short-chain nonionic surfactant is derived from an alcohol of which at least 25% by weight, more preferably at least 50 wt% and most preferably at least 75 wt%, has an alkyl chain length below C_{12}. Advantageously the surfactant system may be substantially free of any nonionic surfactant material having a chain length of C_{12} or above.

Especially preferred are ethoxylated alcohol nonionic surfactants having an average alkyl chain length from C_9 to $C_{11.5}$.

The average number of ethylene oxide groups per mole of alcohol in the short-chain nonionic surfactant is preferably 8 or less, more preferably from 2 to 6.5, and advantageously from 2.5 to 6.

The short-chain nonionic surfactant may consist wholly of straight-chain material, or may contain branched-chain material. Branched chain nonionic surfactants may advantageously give faster dissolution rates than their wholly straight chain counterparts.

It is preferred if the level of free alcohol in the short-chain nonionic surfactant is as low as possible: preferably less than 5 wt% and more preferably less than 1 wt% of the total nonionic surfactant.

Details of some short-chain nonionic surfactants suitable for use in the present invention are given below (* denotes Trade Mark). Mixtures of these materials may also be used in order to achieve intermediate degrees of ethoxylation.

Dobanol* 91 series ex Shell

Ethoxylates:	Dobanol 91-2.5	2.5 EO
	Dobanol 91-5	5 EO
	Dobanol 91-6	6 EO
	Dobanol 91 4-6	4-6 EO

Nominal description: C_{9-11} alcohol with 20-25% branching (C_1-C_4).
Average chain length: 10.14
Chain length distribution:

C_8	linear	0.7	0.7
C_9	linear	17.5	19.0
	C_8 2-methyl	1.0	
	C_7 2-ethyl	0.3	
	C_6 2-propyl	0.2	
C_{10}	linear	40.7	45.8
	C_9 2-methyl	2.9	
	C_8 2-ethyl	1.0	
	other branched	1.2	
C_{11}	linear	25.5	33.3
	C_{10} 2-methyl	2.4	
	C_9 2-ethyl	1.0	
	Other branched	4.4	
C_{12}	linear	0	1.6
	branched	1.6	

Lialet* 111 series ex Enichem

Ethoxylates:	Lialet 111-4	4 EO
	Lialet 111-5	5 EO
	Lialet 111-6	6 EO
	Lialet 111-4-6	4-6 EO
	Lialet 111 6.9	6.9 EO

Nominal description: C_{11} alcohol with 50-60% branching (C_1-C_4).
Average chain length: 11.0
Chain length distribution:

C_{11}	linear	49.2	96.10
	C_{10} 2-methyl	17.3	
	C_9 2-ethyl	9.3	
	C_8 2-propyl	9.7	
	C_7 2-butyl and C_6 2-pentyl	10.6	

Vista* (Alfonic*) series ex Vista Chemicals

Ethoxylates:	Vista 1012-62	6.25 EO
	Vista 1012-52	4.3 EO
	Vista Novel II 1012-52	4.5 EO (narrow range)

Nominal description: C_{10-12} linear alcohol
Average chain length: 10.20
Chain length distribution:

C_{10}	linear	90.0
C_{12}	linear	10.0

Other short-chain nonionic surfactants

The following materials are also suitable for use in the present invention:

Acropol* 91 4-6 ex Exxon:
C_{9-11} chain, 35% branching (C_{1-4}), 4-6 EO

Dobanol* 111 series ex Shell:
98.5% C_{11}, with traces of C_{10} and C_{12}

Synperonic* 91-4-6 ex ICI:
C_{9-11} chain, 60% branching (C_1), 4-6 EO

Lialet* 91 4-6 ex Enichem:
C_{9-11} chain, 60% branching (C_{1-4}), 4-6 EO

Inbentin* $C_{10}E_4$ ex Kolb:
C_{10} linear chain, 4 EO

Longer-chain commercial nonionic surfactants which are preferably absent from the tablets of the invention include the following materials:

Coconut-based materials such as the Lorodac* series ex DAC Chemicals:
C_{12}-C_{16}, average chain length 12.75

Synperonic* nonionics ex ICI, eg Synperonic A3 (3EO):
C_{13-15}, average chain length 13.65:

C_{13}	linear	44.0	67.2
	C_{12} 2-methyl	11.9	
	C_{11} 2-ethyl	3.8	
	C_{10} 2-propyl	3.1	
	C_9 2-butyl and		
	C_8 2-pentyl	4.4	
C_{15}	linear	20.9	35.1
	C_{14} 2-methyl	2.4	
	C_{13} 2-ethyl	1.0	
	C_{12} 2-propyl	0.8	
	C_{11} 2-butyl and		
	C_{10} 2-pentyl and		
	C_9 2-hexyl	2.4	

Dobanol* 23 materials ex Shell (C_{12-13} with 18.1% branching):

C_{12}	38.4
C_{13}	58.9
C_{14}	1.2

Dobanol* 25 materials ex Shell (C_{12-15} with 22.9% branching):

C_{12}	19.9
C_{13}	31.2
C_{14}	29.4
C_{15}	19.1

Dobanol* 45 materials ex Shell (C_{14-15} with 14.8% branching):

C_{14}	60.3
C_{15}	37.5

Incorporation of nonionic surfactant in the tablets

The nonionic surfactants may be concentrated in discrete domains as disclosed in EP 466 485A (Unilever). Since the nonionic detergent compounds are generally liquids, these domains are preferably formed from particulate carrier material impregnated by the nonionic detergent-active compound. Suitable carrier materials include zeolite; zeolite granulated with other materials, for example Wessalith CS (Trade Mark), Wessalith CD (Trade Mark), Vegabond GB (Trade Mark); sodium perborate monohydrate; Burkeite (a spray-dried sodium carbonate/sodium sulphate double salt) as disclosed in EP 221 776A (Unilever).

Other so-called nonionic detergent compounds which may be present in the tablet such as long-chain tertiary amine oxides, tertiary phosphine oxides, and dialkyl sulphoxides.

Nonionic detergent-active compounds may be present in the tablet at an amount from 2 to 50 wt%, preferably from 5 to 30 wt%.

Other detergent-active compounds

The tablets of the invention may contain other surfactants, provided that longer-chain ethoxylated nonionic surfactants are not present in such amounts that the average alkyl chain length of the total nonionic surfactant rises to C_{12} or above.

The total amount of detergent-active material in the tablet of the invention is suitably from 5 to 50% wt%, preferably from 5 to 30 wt%.

Detergent-active material present other than the nonionic surfactants may be other anionic (soap or non-soap), cationic, zwitterionic, amphoteric, or any combination of these.

Anionic detergent-active compounds may be present in an amount of from 0 to 40 wt%, preferably from 0 to 20 wt%. It is preferred if the ratio of nonionic surfactant to anionic surfactant is within the range of 2:8 to 9:1.

Synthetic anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C8-C15; primary and secondary alkyl sulphates, particularly sodium C12-C15 primary alcohol sulphates, olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinate; and fatty acid ester sulphonates.

It may also be desirable to include one or more soaps of fatty acids. These are preferably sodium soaps derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil.

A preferred embodiment of the invention contains alkyl sulphate, preferably primary alkyl sulphate (PAS), as the anionic surfactant. Branched PAS, for example as described in EP 439 316A (Unilever), may be preferred.

An especially preferred embodiment of the invention contains a PAS adjunct wherein the PAS is on a carrier as described previously for nonionic surfactants.

Detergency builders

The detergent tablets of the invention contain one or more detergency builders, suitably in an amount of from 5 to 80 wt%, preferably from 20 to 60 wt%.

The invention is of especial relevance to tablets derived from detergent compositions containing alkali metal aluminosilicates and/or alkali metal carbonate as builders, since such tablets appear to have a particular tendency to harden and so exhibit disintegration and dispersion problems.

Alkali metal (preferably sodium) aluminosilicates may suitably be incorporated in amounts of from 5 to 60% by weight (anhydrous basis) of the composition, and may be either crystalline or amorphous or mixtures thereof, having the general formula:

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

These materials contain some bound water and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiO2 units (in the formula above). Both the amorphous and the crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

Suitable crystalline sodium aluminosilicate ion-exchange detergency builders are described, for example, in 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.

The zeolite may be the commercially available zeolite 4A now widely used in laundry detergent powders. However, according to a preferred embodiment of the invention, the zeolite builder incorporated in the compositions of the invention is maximum aluminium zeolite P (zeolite MAP) as described and claimed in EP 384 070A (Unilever). Zeolite MAP is defined as an alkali metal aluminosilicate of the zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably within the range of from 0.90 to 1.33, and more preferably within the range of from 0.90 to 1.20.

Especially preferred is zeolite MAP having a silicon to aluminium ratio not exceeding 1.07. The calcium binding capacity of zeolite MAP is generally at least 150 mg CaO per g of anhydrous material.

In the tablets of the present invention, the use of zeolite MAP gives two particular advantages: it is a more effective builder than zeolite 4A, and, quite independently, it enables higher total surfactant levels, and more nonionic-rich surfactant systems, to be incorporated without processing problems.

The use of zeolite MAP as a carrier for liquid detergent ingredients is described and claimed in EP 521 635A (Unilever).

Preferred zeolite MAP for use in the present invention is especially finely divided and has a d_{50} (as defined below) within the range of from 0.1 to 5.0 microns, more preferably from 0.4 to 2.0 microns and most preferably

from 0.4 to 1.0 microns. The quantity "d_{50}" indicates that 50 wt% of the particles have a diameter smaller than that figure, and there are corresponding quantities "d_{80}", "d_{90}" etc. Especially preferred materials have a d_{90} below 3 microns as well as a d_{50} below 1 micron.

Alkali metal carbonates may also be suitably incorporated into the tablets in amounts from 0 to 60%, preferably from 2 to 40 wt%. The preferred alkali metal carbonate is that of sodium. An alternative builder system for use within this invention comprises an alkali metal carbonate/calcite system.

Other builders may also be included in the detergent tablet of the invention if necessary or desired: suitable organic or inorganic water-soluble or water-insoluble builders will readily suggest themselves to the skilled detergent formulator. Inorganic builders may be present other than the alkali metal carbonates; while organic builders 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; polycarboxylate polymers; and organic precipitant builders such as alkyl- and alkenylmalonates and succinates, and sulphonated fatty acid salts.

Especially preferred supplementary builders are polycarboxylate polymers, more especially polyacrylates and acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, especially from 1 to 10 wt%; and monomeric polycarboxylates, more especially citric acid and its salts, suitably used in amounts of from 3 to 35 wt%, more preferably from 5 to 30 wt%.

Preferred tabletted compositions of the invention preferably do-not contain more than 5 wt% of inorganic phosphate builders, and are desirably substantially free of phosphate builders. However, phosphate-built tabletted compositions are also within the scope of the invention.

Other ingredients

Fully formulated laundry detergent compositions in accordance with the present invention may additionally contain any suitable ingredients normally encountered, for example, inorganic salts such as sodium silicate or sodium sulphate; antiredeposition aids such as cellulose derivatives and acrylate or acrylate/maleate polymers; fluorescers; bleaches, bleach precursors and bleach stabilisers; proteolytic and lipolytic enzymes; dyes; coloured speckles; perfumes; foam controllers; fabric softening compounds.

As well as the functional detergent ingredients listed above, there may be present various ingredients specifically to overcome difficulties that occur when tabletting, examples of such ingredients are; binders, lubricants and disintegrants.

Tablet lubricants include calcium, magnesium and zinc soaps (especially stearates), talc, glyceryl behenate, Myvatex (Trade Mark) TL ex Eastman Kodak, sodium benzoate, sodium acetate, polyethylene glycols, and colloidal silicas (for example, Alusil (Trade Mark) ex Crosfield Chemicals Ltd).

Examples of disintegrants include organic materials such as starches, for example, corn, maize, rice and potato starches and starch derivatives, such as Primojel (Trade Mark) carboxymethyl starch and Explotab (Trade Mark) sodium starch glycolate; celluloses and cellulose derivatives, for example, Courlose (Trade Mark) and Nymcel (Trade Mark) sodium carboxymethyl cellulose, Ac-di-Sol (Trade Mark) cross-linked modified cellulose, and Hanfloc (Trade Mark) microcrystalline cellulosic fibres; and various synthetic organic polymers, notably polyethylene glycol and crosslinked polyvinyl pyrrolidone, for example, Polyplasdone (Trade Mark) XL or Kollidon (Trade Mark) CL, bentonite clay, citric acid (preferred), malic acid or tartaric acid, in combination with alkali metal carbonate or bicarbonate.

Tablet binders are well known in the art and include natural gums (form example acacia, tragaenth) and sugars (for example glucose, sucrose). As indicated previously, some ingredients may give both functional wash benefits and tabletting benefits.

Tablet forms

The detergent tablet of the invention may be, and preferably is, formulated for use as a complete heavy-duty fabric washing composition.

The powder used to form the tablets by compaction may contain ingredients having different mean particle sizes and distributions, but preferably the starting powder should have an initial granule size in the range 75-1500 micrometres. Depending on the tabletting compaction pressure employed, the smaller particle sizes give smoother tablets with lower porosities whereas the larger particle sizes give tablets with a granular appearance and higher porosities. It is preferred, with the larger particle sizes, to have present a binder which enables the tablet to be formed at lower compaction pressures.

Although one tablet may contain sufficient of every component to provide the correct amount required

for an average washload, it is convenient if each tablet contains a submultiple quantity of the composition required for average washing conditions, so that the consumer may vary the dosage according to the size and nature of the washload. For example, tablet sizes may be chosen such that two tablets are sufficient for an average washload; one or more further. tablets may be added if the washload is particularly large or soiled; and one only tablet may be used if the load is small or only lightly soiled.

Alternatively, larger subdivisible tablets representing a single or multiple dose may be provided with scorings or indentations to indicate unit dose or submultiple unit dose size to the consumer and to provide a weak point to assist the consumer in breaking the tablet if appropriate.

The weight of the tablet will suitably range from 10 to 160 g, preferably from 20 to 50 g, depending on the wash conditions under which it is intended to be used, and whether it represents a single dose, a multiple dose or a submultiple dose.

The tablet may be of any suitable shape, but for manufacturing and packaging convenience is preferably of uniform cross-section, for example, circular or rectangular.

The tablet of the invention may be homogeneous, or may consist of more than one discrete region: for example, two or more layers of different composition may be present, or a core region may be wholly surrounded by an outer region of different composition.

The diameter of the tablet will suitably range from 1 to 10 cm, preferably from 2 to 6 cm.

Tabletting

As previously indicated, the tablets of the invention are prepared by compaction of a particulate detergent composition. The preferred bulk density of the detergent powder is at least 700 g/litre, more preferably 800 g/litre. Any suitable. tabletting apparatus may be used.

For any given starting composition, the disintegration time will vary with the compaction pressure used to form the tablet. If the compaction pressure is too low, the tablet will tend to crumble and break up in the dry state, on handling and packaging; an increase in compaction pressure will improve tablet integrity, but eventually at the expense of disintegration time in the wash liquor.

Using an Instron (Trade Mark) Universal Testing Machine at constant speed, or a Research and Industrial screw hand press, to operate a steel punch and die, it has been found that effective tablets may be produced using compaction pressures ranging from 35 to 1000 Ncm^{-2}, especially from 75 to 500 Ncm^{-2}.

The optimum compaction pressure will depend to some extent on the starting composition; for example, a formulation containing a high proportion of organic ingredients (for example, surfactants) and a low proportion of inorganic salts may require a compaction pressure lower than that required for a formulation containing a lower proportion of organic ingredients and a higher proportion of inorganic salts; and a dry-mixed formulation will generally required a higher pressure than will a spray-dried powder.

As a measure of the resistance of the tablets to fracture, the diametral fracture stress σ_o calculated from the following equation:

$$\sigma_o = \frac{2P}{Dt}$$

where σ_o is the diametral fracture stress (Pascals), P is the applied load to cause fracture (Newtons), D is the tablet diameter (metres) and t is the tablet thickness (metres).

Tablets of the invention preferably have a diametral fracture stress of at least 5 kPa, and more preferably at least 7 kPa.

EXAMPLES

The following non-limiting examples illustrate the invention. Parts and percentages are by weight unless otherwise stated. Examples identified by numbers are in accordance with the invention while those identified by letters are comparative.

In the Examples, the following tests were used to assess the dissolution properties of the tablets.

1 Rotating Cage Test

Preweighed tablets were placed in a cage of perforated metal gauze (7 cm x 7 cm x 6 cm) having apertures about 5mm square per cm^2. The cage was then suspended in a beaker of demineralised water at 23°C and rotated at 80 rpm. The mass of tablet dissolved was calculated by a standard conductance procedure which involved measuring the conductance of the water at a given time and comparing this reading with that obtained from a fully dissolved powder of identical formulation and weight.

It is preferable if the tablet is capable of dissolving to an extent of 60% by weight in 15 minutes. More preferably the tablet will be capable of dissolving to an extent of 75% by weight in 15 minutes.

2 Machine Test A

The dissolution of the tablets was studied in a Miele W756 front-loading automatic washing machine. The tablet was placed in the drum of the machine, the machine was programmed on the economy main wash with a cold water fill (10.5 litres demineralised water, isothermal at 11°C). No load was present. The percentage of the tablet dissolved was calculated using the standard conductance procedure described above.

3 Machine Test B

Tablets were placed in an experimental perspex washing machine. The programme of the washing machine was based upon the cotton cycle of the Spanish Zanussi (using 10 litres of cold Wirral water). The times taken for 50% and 90% of the tablet to dissolve were recorded. The standard conductance test as described above was used to calculate the weight of tablet dissolved.

Examples 1-4 and Comparative Examples A-D

Detergent base powders were prepared to the following general formulation:

Base powder	wt%
Zeolite	51.2
Sodium citrate	8.5
Water	14.8
Postdosed	
Nonionic surfactant	25.5

The base powders were prepared by spray-drying. The nonionic surfactant was then stirred into the base powder and fluid bed mixed for 10 minutes at 70°C, and the powders were then allowed to weather. The tablets were produced using the Instron Universal Testing Machine at constant speed. Tablets so produced had end fracture strengths of 5 or 15 kPa and respectively thicknesses of 12.2 mm or 10.8 mm.
The nonionic surfactants used were as described below:

Example	Nonionic surfactant
Examples A and B	Synperonic A7 (ICI)
Examples C and D	Coco 6.5 EO (Kolb)
Examples 1 and 2	Dobanol 91-6 T (Shell)
Examples 3 and 4	Alfonic 1012-62 (Vista)

Details of these nonionic surfactants have been given earlier in the text.
Dissolution results using the rotating cage test described previously are set out in Table 1.

Table 1

Example	Fracture	Weight Tablet Dissolved (%) after			
	Stress	3 min	7 min	11 min	15 min
A	5 kPa	9	19	35	49
B	15 kPa	6	18	34	43
C	5 kPa	13	30	57	72
D	15 kPa	7	29	42	50
1	5 kPa	58	83	97	100
2	15 kPa	36	59	73	85
3	5 kPa	55	82	97	100
4	15 kPa	30	58	71	82

It must be appreciated that the rotating cage experiment is a rigorous test, as mechanical agitation is minimal and dissolution is mainly due to physico-chemical effects.

A more realistic approach is to study the dissolution in a washing machine, using Machine Test A described previously.

Table 2

Example	Weight tablet dissolved (%) after (min)					
	0.5	1	2	3	4	5
A	32.8	70.2	83.8	88.1	90.2	91.9
B	35.6	61.4	75.6	80.5	82.9	84.8
C	30.8	66.3	83.3	88.6	91.4	92.7
D	37.9	58.4	77.8	83.1	86.6	89.3
1	30.7	68.6	86.6	90.9	92.6	93.6
2	35.2	62.4	83.3	90.0	93.7	95.1
3	34.2	67.4	87.7	93.1	94.8	95.5
4	34.2	64.8	84.1	90.8	93.9	96.4

Examples 5-8 and Comparative Examples E and F

High-bulk-density granular detergent compositions were prepared to the following formulations:

Base Powders	Examples E and F	Examples 5 and 6	Examples 7 and 8
Synperonic A7	15.8	-	-
Dobanol 91-6	-	15.8	-
Dobanol 91-3.75**	-	-	15.8
Zeolite 4A	31.8	31.8	31.8
Sodium citrate	5.3	5.3	5.3
Water	9.2	9.2	9.2
Post-dosed			
Di-silicate	2.5	2.5	2.5
Sodium carbonate	9.7	9.7	9.7
Antifoam granules	2.5	2.5	2.5
TAED	7.8	7.8	7.8
Sodium perborate mono	15.0	15.0	15.0
Calcium EDTMP	0.4	0.4	0.4
Total	100.0	100.0	100.0

** Dobanol 91-3.75 is a mixture of Dobanol 91-6T and Dobanol 91-2.5 at a ratio of 3.57:6.43.

The base powders were produced by spray-drying, and further ingredients were post dosed. Tablets were produced using the Instron Universal Testing Machine at constant speed. The tablets had fracture strengths of 5 kPa and 15 kPa with tablet thicknesses of 18.5 mm and 16.6 mm respectively.

The tablets were placed in a Miele W756 washing machine and the amount of tablet dissolution measured using Machine Test A. The results are shown in Table 3.

Table 3

Example	Fracture	Amount tablet dissolved (%) after			
	Stress	0.5 min	1 min	3 min	5 min
E	5 kPa	19	46	83	91
F	15 kPa	16	31	56	66
5	5 kPa	20	51	94	98
6	15 kPa	17	36	67	80

The tablets containing Dobanol 91.6T, a short chain nonionic, dissolved faster than the tablet containing

Synperonic A7, a longer chain nonionic, at both high and low fracture strengths.

After 8 minutes the machine cycle was stopped and the residue of the tablet removed from the drum, dried and weighed. This procedure was repeated three times for each tablet. The mean residue was calculated as a percentage of the original tablet's weight. The results are shown in Table 4.

Table 4

Example	Mean residue % (%) of original weight)
E	1.3
F	32.3
7	0
8	7.3

The results clearly show that the dissolution and disintegration of tablets containing short chain nonionic surfactants was faster than those containing longer chain nonionic surfactants.

Example 9 and Comparative Example G

Spray-dried powders were made to the following formulations:

	Example G	Example 9
Base powder	wt%	wt%
Zeolite 4A	30	30
Sodium citrate	5	5
Synperonic A7	10.22	0.22
Dobanol 91-5	-	10.00
Water	8.7	8.7
Postdosed		
Perborate monohydrate	14	14
TAED (83%)	7.4	7.4
Calcium EDTMP (33%)	0.37	0.37
Antifoam granule	3.0	3.0
Savinase	1.1	1.1
Sodium carbonate	13.0	13.0
PAS noodles	6.7	6.7
	100	100

* 89-90% Coconut PAS (Empicol LZV/E Trade Mark ex Albright & Wilson), as described in EP 466 485A (Unilever).

Tablets were manufactured on the Instron using the 45mm diameter die and 45g of powder.

Dissolution rates were measured by Machine Test B described previously, and were as shown in Table 5.

Table 5

Example	Fracture Stress (kPa)	Time for 50% to dissolve (min)	Time for 90% to dissolve (min)
G	16.3	4.75	13
9	16.2	3.75	8.5

Table 5 demonstrates that short chain nonionic surfactants improve dissolution rates in a mixed surfactant system.

Examples 10 and 11

Tablets were compacted from detergent compositions as described below, at a compaction pressure of 15 kPa.

	Example 10 (wt%)	Example 11 (wt%)
Base powder		
Dobanol 91-6T	11.1	-
Dobanol 91-4T	-	11.1
Zeolite 4A	31.8	31.8
Sodium citrate	5.3	5.3
Water	9.2	9.2
PAS adjunct*	4.7	4.7
Postdosed		
Di-silicate	2.5	2.5
Sodium carbonate	9.7	9.7
Antifoam granule	2.5	2.5
Sodium perborate monohydrate	15.0	15.0
TAED	7.8	7.8
Calcium EDTMP	0.4	0.4
	100.0	100.0

* - PAS adjunct:	wt%
PAS	38
Zeolite	56
Sodium carbonate	4
Water	2

Machine Test A, as described previously, was used to determine dissolution properties. The results, compared with Comparative Example B (see previously), are shown in Table 6.

Table 6

	Mean Residue (% Weight of original tablet)
Example B	39.7
Example 10	7.4
Example 11	9.2

It is again demonstrated that even in the presence of PAS adjunct the presence of a short chain nonionic decreases the dissolution rate and so decreases the residue of tablet in the machine drum.

Example 12

Tablets were prepared by compaction of a high bulk density detergent powder having the following formulation (in weight %):

Nonionic base granule	
Dobanol 91-6T	8.3-8.9
Zeolite 4A	28.2-29.6
Sodium carbonate	14.2-15.1
Acrylic/maleic copolymer	4.3-4.6
SCMC	0.5
Fluorescers	0.21
Polyethylene glycol 1500	2.4-6.0
Moisture	7.0-7.4

PAS adjunct	
(see earlier Examples)	5.7

Postdosed ingredients	
Sodium perborate monohydrate	14.0
TAED granules	7.4
Calcium EDTMP	0.4
Antifoam granules	3.0
Perfume	0.5
Protease granules	0.8
Lipolase granules	0.2

The method of preparation of the powder was as follows. A nonionic base granule was prepared by granulating the sodium carbonate (light ash) and zeolite 4A with the Dobanol 91-6T, aqueous polyacrylic acid, SCMC and fluorescers in a batch high-speed mixer/granulator (Fukae (Trade Mark) FS100), followed by drying in a batch fluid bed dryer, screening to a 250-1700 micron fraction.

The dried granular material was coated with the polyethylene glycol 1500 by spraying it on as a melt, from a pressurised kettle maintained at 60-70°C, in a batch rotary mixer, and screened again to remove oversize material.

The PAS adjunct, which had the formulation given in Examples 10 and 11, was prepared by dry neutralisation: PAS acid was sprayed onto zeolite and carbonate and neutralised in situ by the carbonate. This too was screened to 250-1700 micrometres.

The nonionic base granule, the PAS adjunct and the remaining ingredients were mixed together in a batch rotary mixer.

Tabletting was carried out using the Manesty single punch eccentric press (Thomson and Capper, Runcorn, Cheshire, England). The tablets, of thickness 45 mm and diameter 29.1-29.3 mm and each containing 50 g of powder, generally had fracture strengths of 10-13 kPa.

The tablets exhibited excellent dissolution characteristics and cleaning performance.

Examples 13 and 14, Comparative Examples H and J

In this experiment, mixed nonionic surfactant systems having different average chain lengths were used.

25 g and 50 g tablets were prepared, as in earlier Examples, by compacting high bulk density detergent powders. Nonionic surfactants were adsorbed onto Wessalith (Trade Mark) CD, a commercial zeolite 4A/polyacrylate granulated carrier material ex Degussa, at a level of 25.5 wt%, and the postdosed ingredients used in Examples 5-8 (37.9 wt%) admixed. The tablets were prepared to a strength of 15 kPa.

The nonionic surfactants used were as follows:

Example	Nonionic surfactant	Average chain length	Average EO
H	Synperonic A7	13.7	7.0
J	Synperonic A7 (43 parts)) Synperonic A3 (57 parts))	13.7	4.3
13	Synperonic A7 (49 parts)) Dobanol 91-2.5 (51 parts))	11.4	4.0
14	Synperonic A7 (25 parts)) Dobanol 91-6T (75 parts))	10.3	6.2

The tablets (2 x 50 g or 4 x 25 g) were used in a 40°C wool wash (low agitation) in the Miele machine, with a 1.4 kilo clean load. The water inlet temperature was 20°C. The tablets were dosed in a dispensing device of the type used for high bulk density powders. The following residues (g) were found in the dispensing device at the end of the main wash (each result being the mean of three values):

H	4 x 25 g	36.1
	2 x 50 g	43.1
J	4 x 25 g	26.0
	2 x 50 g	31.4
13	4 x 25 g	4.2
	2 x 50 g	13.0
14	4 x 25 g	9.9
	2 x 50 g	26.7

These results show the benefit of reducing the average alkyl chain length of the nonionic surfactant system. The benefit is especially marked with the smaller (25 g) tablets.

In Machine Test A (the economy wash), in which the agitation is high, the following residues (g) were found in the drum of the Miele machine after 5 minutes, were obtained, each result being the mean of three values:

4 x 25 g	6.4
2 x 50 g	11.8
4 x 25 g	2.2
2 x 50 g	2.3

Thus the benefits of using short-chain nonionic surfactant are also observed in a high-agitation wash.

Examples 15 and 16, Comparative Example K

Tablets were prepared, as generally described in Example 12, to the following formulations (in parts by weight), the sodium perborate, TAED, calcium EDTMP and antifoam granules being postdosed:

	K	15	16
CocoPAS	5.7	5.7	1.2
Nonionic surfactant:			
Synperonic A7	9.0	-	-
Dobanol 91-6T	-	9.0	10.5
Zeolite 4A	31.2	31.2	35.2
Sodium carbonate	14.8	14.8	17.1
Polyethylene glycol 1500	3.9	3.9	3.9
Acrylic/maleic copolymer	4.6	4.6	4.6
Sodium perborate monohydrate	14.0	14.0	14.0
TAED	7.4	7.4	7.4
Calcium EDTMP	0.4	0.4	0.4
Antifoam granules	3.0	3.0	3.0
Moisture	7.7	7.7	7.7

The tablets were prepared to strengths ranging from 9 to 14 kPA, using the Manesty machine used in Example 12.

The tablets showed the following properties, test methods being as described in earlier Examples:

	Tablet strength	T_{90} in economy wash	Residue in wool wash (g)
K	9 kPa	3.5-5.5	5-20 g
15	12 kPa	3.5-5.0	5-10 g
16	14 kPa	3.5-5.0	5-10 g

Claims

1 A tablet of compacted detergent powder comprising a surfactant system comprising a nonionic surfactant, at least one detergency builder and optionally other detergent ingredients, characterised in that the nonionic surfactant comprises a condensation product of ethylene oxide with an aliphatic alcohol having an average alkyl chain length of less than C_{12}.

2 A detergent tablet according to claim 1, characterised in that at least 50% by weight of the alcohol from which the nonionic condensation product is derived has an alkyl chain length below C_{12}.

3 A detergent tablet according to claim 2, characterised in that at least 75% by weight of the alcohol from which the nonionic condensation product is derived has an alkyl chain length below C_{12}.

4 A detergent tablet according to any preceding claim, characterised in that it is substantially free of ethoxylated nonionic surfactant having an alkyl chain length of C_{12} or above.

5 A detergent tablet according to any preceding claim, characterised in that the nonionic condensation product has an average alkyl chain length within the range of from C_9 to $C_{11.5}$.

6 A detergent tablet according to any preceding claim, characterised in that the average degree of ethoxylation of the condensation product is 8 or less.

7 A detergent tablet according to claim 6, characterised in that the average degree of ethoxylation of the nonionic condensation product is within the range of from 2 to 6.5.

8 A detergent tablet according to claim 7, characterised in that the average degree of ethoxylation of the nonionic condensation product is within the range of from 2.5 to 6.

9 A detergent tablet according to any preceding claim, characterised in that the surfactant system further comprises anionic surfactant.

10 A detergent tablet according to claim 9, characterised in that the anionic surfactant comprises primary alkyl sulphate.

11 A detergent tablet according to any preceding claim characterised in that it is the compaction product of a particulate composition having an initial bulk density of at least 700 g/litre.

12 A detergent tablet according to any preceding claim, characterised in that it comprises from 5 to 80 wt% (anhydrous basis) of an alkali metal aluminosilicate.

13 A detergent tablet as claimed in any preceding claim, characterised in that it is capable of dissolving to an extent of 60% by weight in 15 minutes in water at 23½C in the rotating cage dissolution test as herein described.

14 Use of a nonionic surfactant which is a condensation product of ethylene oxide with an aliphatic alcohol having an average alkyl chain length of less than C_{12} to improve the disintegration and dissolution in the wash liquor of a tablet of compacted detergent powder.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 93 30 9120

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)
A	EP-A-0 482 627 (KAO CORPORATION) * page 2, line 40 - page 4, line 22 *	1,6,9, 12,14	C11D17/00 C11D1/72
A	EP-A-0 481 793 (UNILEVER PLC) * page 2, line 46 - page 4, line 51 *	1,6,9, 10,12,14	
A	EP-A-0 355 626 (HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN) * page 2, line 22 - page 3, line 32 * * page 5, line 4 - page 5, line 31; example 1 *	1,9,11, 12,14	

TECHNICAL FIELDS SEARCHED (Int.Cl.5)

C11D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	22 February 1994	Doolan, G

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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