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(54) **PROCESS FOR PRODUCTION OF UNI-CORE DETERGENT PARTICLES**

(57) The present invention relates to a method for producing uni-core detergent particles capable of producing uni-core detergent particles containing an anionic surfactant in a high yield, wherein the uni-core detergent particles are generally very low in skin irritability, and fa-

vorable in biodegradability, and inhibited in the particle growth, and have a sharp particle size distribution, without necessitating a drying step for removing the water after the granulation step.

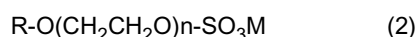
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DescriptionTECHNICAL FIELD

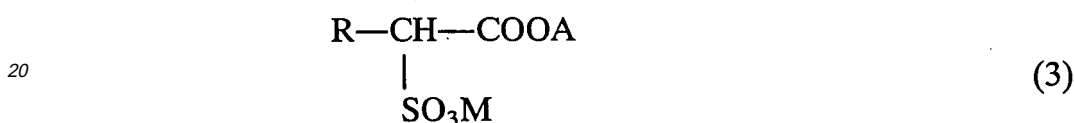
5 **[0001]** The present invention relates to a method for producing uni-core detergent particles containing, as an anionic surfactant, a compound represented by any of the formulae (1) to (3):



10 wherein R is an alkyl group or an alkenyl group having 10 to 18 carbon atoms; and M is an alkali metal atom or an amine,



15 wherein R is an alkyl group or an alkenyl group having 10 to 18 carbon atoms; n is an average number of moles added of from 0.1 to 3.0; and M is an alkali metal atom, or an ammonium or an organic amine, and



25 wherein R is an alkyl group or an alkenyl group having 4 to 22 carbon atoms; M is an alkali metal atom, an alkaline earth metal atom, an alkanolamine or an ammonium; and A is an alkyl group having 1 to 4 carbon atoms, H, or M.

BACKGROUND ART

30 **[0002]** One of the methods for producing detergent particles includes a production method including the step of mixing a powdery substance and a liquid surfactant composition. Among them, there are so far various disclosures of powdery detergents in which an anionic surfactant represented by the above-mentioned formula (1) is formulated as a detergent surfactant for the purpose of improvements in high detergent activation ability, re-deposition preventing ability, environmental friendliness, and solvency by a combination of surfactants, and the like.

35 **[0003]** For example, a method for producing a granular detergent composition using a liquid surfactant composition composed of an anionic surfactant represented by the above-mentioned formula (1), a nonionic surfactant, and water (Patent Publication 1); and a production method including the step of formulating an anionic surfactant represented by the above-mentioned formula (1) in a detergent slurry (Patent Publication 2), or the step of adding an anionic surfactant represented by the above-mentioned formula (1) to an intermediate product of the extrusion-molding (Patent Publication 3) are disclosed.

40 **[0004]** However, when the detergent particles are produced by the method of Patent Publication 1, it is necessary to previously neutralize an alkyl sulfate which is poor in stability as compared to LAS or the like in a nonionic surfactant, so that there is yet a concern from the aspect of stability of the anionic surfactant represented by the above-mentioned formula (1).

45 **[0005]** In addition, while the production methods of Patent Publications 2 and 3 are free from any problems in the aspect of stability of the anionic surfactant represented by the above-mentioned formula (1), the dissolubility yet remains unsatisfied because both of the resulting detergent particles go through the treatment of increasing compactness.

50 **[0006]** In addition, Patent Publication 4 discloses a method for producing a granular detergent composition including the steps of oil-absorbing a paste of the anionic surfactant represented by the above-mentioned formula (2) to silica or a silicate, granulating the mixture, and drying the granules. The production method as described above has an advantage that the anionic surfactant can be formulated in a high content. On the other hand, in order to facilitate the production of the granular detergent composition as described above, an oil-absorbing carrier such as silica or a silicate is necessary, and further a drying step is necessitated after the granulation step in order to remove water contained in the above-mentioned paste.

55 **[0007]** In addition, Patent Publication 5 discloses a method for producing a detergent composition including the step of mixing a surfactant composition containing an anionic surfactant represented by the above-mentioned formula (2), a nonionic surfactant, and water, with an adsorbent powder. However, in this production method, it is impossible to prepare a free-flowable powder detergent in a high yield by a method including the step of mixing the surfactant composition in a paste-like form with water-soluble powder detergent particles.

[0008] In addition, a method for producing a high-bulk density detergent composition including the steps of making an anionic surfactant represented by the above-mentioned formula (3) in the form of a powder, powder-blending the anionic surfactant with an alkali builder, concurrently adding a water-containing binder thereto, and granulating the mixture (Patent Publication 6); and a method for producing a high-bulk density detergent including the steps of concentrating an anionic surfactant represented by the above-mentioned formula (3), and directly formulating the concentrate into a kneading step (Patent Publication 7) are disclosed.

[0009] However, when detergent particles are produced according to the production method of Patent Publications 6 or 7, the dissolubility yet remains unsatisfied because both of the resulting detergent particles go through the treatment of increasing compactness.

Patent Publication 1: JP-A-Hei-6-17098

Patent Publication 2: JP-A-Hei-6-220499

Patent Publication 3: JP-A-Hei-8-504458

Patent Publication 4: WO 0031223

Patent Publication 5: JP-A-Hei-03-62899

Patent Publication 6: JP-A-Hei-4-359098

Patent Publication 7: JP-A-Hei-9-143500

DISCLOSURE OF INVENTION

PROBLEMS TO BE SOLVED BY THE INVENTION

[0010] Therefore, an object of the present invention is to provide a method for producing uni-core detergent particles including the step of formulating the anionic surfactant represented by any of the above-mentioned formulae (1) to (3), in which the method for producing the detergent particles secures the stability of the anionic surfactant represented by the above-mentioned formulae (1) to (3), and provides excellent dissolubility.

MEANS TO SOLVE THE PROBLEMS

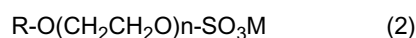
[0011] Specifically, the gist of the present invention relates to a method for producing uni-core detergent particles having an average particle size of 150 μm or more and a degree of particle growth of 1.5 or less, including the steps of:

step A): preparing a surfactant composition containing:

a) an anionic surfactant represented by any of the following formulae (1) to (3):



wherein R is an alkyl group or an alkenyl group having 10 to 18 carbon atoms; and M is an alkali metal atom or an amine,



wherein R is an alkyl group or an alkenyl group having 10 to 18 carbon atoms; n is an average number of moles added of from 0.1 to 3.0; and M is an alkali metal atom, or an ammonium or an organic amine, and



wherein R is an alkyl group or an alkenyl group having 4 to 22 carbon atoms; M is an alkali metal atom, an alkaline earth metal atom, an alkanolamine or an ammonium; and A is an alkyl group having 1 to 4 carbon atoms, H, or M, and

b) water in an amount of from 25 to 65 parts by weight based on 100 parts by weight of the above-mentioned component a);

step B): mixing the surfactant composition obtained in step A) and base particles having a supporting ability of 20 mL/100 g or more and containing a water-soluble inorganic salt produced by spray-drying, while substantially maintaining the form of the base particles; and
 step C): surface-modifying the mixture obtained in step B) with a fine powder.

EFFECTS OF THE INVENTION

[0012] By using the method for producing uni-core detergent particles of the present invention, the effect that the uni-core detergent particles containing an anionic surfactant represented by the above-mentioned formulae (1) to (3), which is generally very low in skin irritability and favorable in biodegradability, the uni-core detergent particles having an inhibitory particle growth, and a sharp particle size distribution can be produced in a high yield, without necessitating a drying step for removing the water after the granulation step is exhibited. By providing a sharp particle size distribution, a detergent which is not only improved in external appearance, but also favorable in free-flowability, and excellent in dissolubility can be obtained.

BEST MODE FOR CARRYING OUT THE INVENTION

[0013] One of the great features of the method for producing uni-core detergent particles of the present invention (hereinafter referred to as the production method of the present invention) resides in that the method, as described above, includes the steps of:

step A): preparing a surfactant composition containing a) the anionic surfactant represented by the above-mentioned formulae (1) to (3), and b) water in an amount of from 25 to 65 parts by weight based on 100 parts by weight of the above-mentioned component a);

step B): mixing the surfactant composition obtained in step A) and base particles having a supporting ability of 20 mL/100 g or more and containing a water-soluble inorganic salt produced by spray-drying, while substantially maintaining the form of the base particles; and

step C): surface-modifying the mixture obtained in step B) with a fine powder.

[0014] By using the production method of the present invention having the above-mentioned feature, the effect that the detergent particles containing the anionic surfactant represented by the above-mentioned formulae (1) to (3), which is generally very low in skin irritability and favorable in biodegradability, the detergent particles having an inhibitory particle growth, and a sharp particle size distribution can be produced, without necessitating a drying step for removing the water after the granulation step is exhibited.

[0015] In the production method of the present invention, a mechanism for exhibiting an effect of not necessitating a drying step for removing the water after the granulation step is considered to be due to the fact that in step B), when the surfactant composition containing an anionic surfactant represented by the formulae (1) to (3) and water contacts with the base particles containing a water-soluble inorganic salt, water in the surfactant composition is taken away by the water-soluble inorganic salt, and the composition of the anionic surfactant represented by the formulae (1) to (3) loses free-flowability, whereby powdering can be carried out without adding the drying step.

[0016] The production method of the present invention will be described more specifically hereinbelow.

[Step A)]

[0017] In the production method of the present invention, step A) is a step of preparing a surfactant composition containing a) an anionic surfactant represented by the above-mentioned formulae (1) to (3), and b) water in an amount of from 25 to 65 parts by weight based on 100 parts by weight of the above-mentioned component a).

[Components in Surfactant Composition]

[0018] As for the component a), in the formula (1), R is an alkyl group or an alkenyl group having 10 to 18 carbon atoms, and preferably 12 to 16 carbon atoms. M is preferably an alkali metal atom such as Na or K, or an amine such as monoethanolamine or diethanolamine, and especially preferably Na or K from the viewpoint of an improvement in detergency of the detergent composition.

[0019] In addition, in the formula (2), R is an alkyl group or an alkenyl group having 10 to 18 carbon atoms, and preferably 12 to 16 carbon atoms. The average number of moles added n is from 0.1 to 3.0, and preferably from 0.1 to 2.0. M is preferably an alkali metal atom such as Na or K, an ammonium or an organic amine such as monoethanolamine or diethanolamine, and Na or K is especially preferable from the viewpoint of an improvement in detergency of the

detergent composition.

[0020] In addition, in the formula (3), R is an alkyl group or an alkenyl group having 4 to 22 carbon atoms; M is an alkali metal atom, an alkaline earth metal atom, an alkanolamine or an ammonium; and A is an alkyl group having 1 to 4 carbon atoms, H, or M.

[Physical Properties of Surfactant Composition]

[0021] It is desired that the surfactant composition has a temperature range in which the viscosity of the surfactant composition is 10 Pa·s or less, and preferably 5 Pa·s or less in an operable temperature range of the surfactant composition, from the viewpoint of handling in the production. It is preferable that the temperature range as mentioned above exists preferably in a range up to 70°C, and more preferably in a range up to 60°C, from the viewpoint of the stability of the surfactant composition. Here, the viscosity is determined with a coaxial double cylindrical rotary viscometer (manufactured by HAAKE; sensor: SV-DIN) at a shearing rate of 50 1/s.

[0022] The surfactant composition prepared in step A) greatly varies in viscosity depending on its water content. It is preferable that a surfactant composition having a desired water content, i.e., a desired viscosity is prepared by adjusting with an amount of water of an alkali compound usable in the preparation of the surfactant composition by neutralizing an acid precursor of the component a) with the alkali compound. It is generally known that when the surfactant composition contains the component a) and water in an amount of from 25 to 65 parts by weight (water content of the surfactant composition is from 20 to 40%) based on 100 parts by weight of the component a), the viscosity is lowered, thereby making its handling easy. It is preferable that the water of the surfactant composition is adjusted within this range in the present invention.

[0023] In addition, since the acid precursor of the component a) is very unstable and more likely to be degraded, it is preferable that the adjustment is made so that the degradation is suppressed. The method of adjustment is not particularly limited, and a known method can be used. For example, the method may be carried out by removing heat of neutralization with a heat exchanger or the like using a loop reactor while cautiously temperature-controlling the acid precursor of the component a) and the surfactant composition. A temperature range during production includes a temperature of from 30° to 60°C, and a temperature range for storage after the production includes a temperature of 60°C or less. In addition, the surfactant composition may be used by optionally elevating the temperature upon use.

[0024] When the anionic surfactant composition represented by the formula (1) or (2) is used, it is preferable that the surfactant composition has an excess alkalinity from the viewpoint of suppressing the degradation. On the other hand, when the anionic surfactant composition represented by the formula (3) is used, a pH is preferably from 4 to 9, and a pH is more preferably from 5 to 8.

[0025] In addition, the adjusted surfactant composition may contain an unreacted alcohol or an unreacted polyoxyethylene alkyl ether upon the production of the acid precursor of the component a), sodium sulfate, which is a by-product of the neutralization reaction, or a pH buffering agent, which can be added during the neutralization reaction, a decolorizing agent, or the like.

[0026] Further, the surfactant composition usable for the present invention may contain a known component ordinarily used in detergents, for example, a surfactant known in the field of laundry detergents; a re-deposition preventing agent such as acrylic acid polymer, acrylic acid-maleic acid copolymer, and carboxymethyl cellulose; a reducing agent such as a sulfite; a fluorescent brightener, or the like.

[0027] Incidentally, the component a) is contained in an amount within the range of preferably from 5 to 30% by weight, and more preferably from 10 to 30% by weight of the uni-core detergent particles obtainable in the present invention, from the viewpoint of an improvement in detergency.

[0028] The component b) is water contained in an amount of from 25 to 65 parts by weight, and preferably from 30 to 50 parts by weight of the surfactant composition, based on 100 parts by weight of the above-mentioned component a).

[Step B)]

[0029] In the present invention, step B) is a step of mixing the surfactant composition obtained in step A) and base particles having a supporting ability of 20 mL/100 g or more and containing a water-soluble inorganic salt produced by spray-drying, while substantially maintaining the form of the base particles.

[0030] In the present invention, one feature resides in that step B) is carried out. In step B), by mixing the surfactant composition with the base particles containing the water-soluble inorganic salt to contact with each other, the loss of free-flowability of the surfactant composition exhibited by taking water in the surfactant composition away by the water-soluble inorganic salt can be utilized.

[Base Particles Containing Water-Soluble Inorganic Salt]

[0031] The base particles usable in step B) have a supporting ability of 20 mL/100 g or more and contain a water-soluble inorganic salt produced by spray-drying.

[0032] The above-mentioned base particles are prepared by spray-drying a slurry containing the water-soluble inorganic salt. The water-soluble inorganic salt is not particularly limited. For example, among the above-mentioned builders generally used in laundry detergents, sodium carbonate, potassium carbonate, sodium sulfate, or the like is preferable.

[0033] As for the base particles, a powder obtained by spray-drying an aqueous slurry properly formulated with, for example, a builder generally used in laundry detergents including, for example, one or more kinds of metal ion capturing agents such as zeolite, citrates and sodium tripolyphosphate; an alkalizing agent such as sodium carbonate or potassium carbonate; one or more kinds of base materials that exhibit both the metal ion capturing ability and the alkalizing ability such as a crystalline silicate; and the like; and/or other base material agent generally usable in detergent compositions including, for example, a surfactant known in the field of laundry detergents, a re-deposition preventing agent such as an acrylic acid polymer, an acrylic acid-maleic acid copolymer or carboxymethyl cellulose, an inorganic powder such as sodium sulfate or a sulfite, a fluorescent brightener, or the like is preferable. In addition, the alkalizing agent may be removed from the base particles when a base material agent or the like to be degraded by contacting with an alkali is contained in the base particles or added in step B), from the viewpoint of suppressing degradation of the base material agents.

[0034] Among them, it is preferable that zeolite is used in combination with the above-mentioned water-soluble inorganic salt. When zeolite is formulated, water in the base particles after the spray-drying is contained in an amount of preferably 5% by weight or less, and more preferably 3% by weight or less of the base particles, from the viewpoint of increasing an action of water absorption in zeolite.

[0035] The base particles in which the water-soluble inorganic salt and zeolite, which are preferably contained in the base particles, are formulated in an amount of 60% by weight or more in total are favorable to take away water of the surfactant composition.

[0036] The conditions upon spray-drying the slurry for preparing the above-mentioned base particles (temperature, spray-drying apparatus, spraying method, drying method, or the like) are not particularly limited, and a known method may be used. The physical properties of the base particles used in the present invention are given hereinbelow.

[Physical Properties of Base Particles]

[0037] The base particles have a supporting ability of 20 mL/100 g or more, and preferably 30 mL/100 g or more. Within this range, the aggregation of the base particles themselves is suppressed, thereby making it favorable to maintain the uni-core owned by the particle in the detergent particles.

[0038] The determination method for the supporting ability is as follows.

A cylindrical mixing vessel of an inner diameter of about 5 cm and a height of about 15 cm which is equipped with agitation impellers in the inner portion thereof is charged with 100 g of a sample. While stirring with the agitation impellers at 350 r/min, linseed oil is supplied at 25°C into the mixing vessel at a rate of about 10 mL/min. The supporting ability is defined as an amount of linseed oil supplied when the agitation torque reaches the highest level.

[0039] The base particles have a bulk density of preferably from 200 to 1000 g/L, more preferably from 300 to 1000 g/L, even more preferably from 400 to 1000 g/L, and especially preferably from 500 to 800 g/L. The bulk density is measured by a method according to JIS K 3362.

[0040] The base particles have an average particle size of preferably from 150 to 500 μm , and more preferably from 180 to 350 μm . The average particle size is calculated by vibrating a sample using standard sieves according to JIS Z 8801 (sieve openings of from 2000 to 125 μm) for 5 minutes, and thereafter determining the median particle size from a weight percentage depending upon the size openings of the sieves.

[Mixing Method]

[0041] It is preferable that a mixer for mixing the surfactant composition and the base particles usable in step B) is, for example, a mixer equipped with a nozzle for adding the surfactant composition or a jacket for controlling the temperature within a mixer.

[0042] As the mixing conditions in step B), mixing conditions are selected such that the base particles substantially maintain their shapes, i.e., the base particles do not undergo disintegration. For example, when a mixer equipped with agitation impellers is used, in a case of a mixer equipped with mixing impellers for the agitation impellers having a paddle shape, the agitation impellers have a Froude number of preferably from 0.5 to 8, more preferably from 0.8 to 4, and even more preferably from 0.5 to 2, from the viewpoint of the suppression of the disintegration of the water-soluble inorganic salt and mixing efficiency. In addition, in a case where the mixing impellers have a screw shape, the agitation impellers

have a Froude number of preferably from 0.1 to 4, and more preferably from 0.15 to 2. Also, in a case where the mixing impellers have a ribbon shape, the agitation impellers have a Froude number of preferably from 0.05 to 4, and more preferably from 0.1 to 2.

[0043] Further, there may be also employed a mixer equipped with agitation impellers and disintegration impellers. When the base particles and the surfactant are mixed by using the mixer, the disintegration impellers have been conventionally subjected to high-speed rotation, from the viewpoint of accelerating mixing. However, in the present invention, it is preferable not to substantially rotate the disintegration impellers, from the viewpoint of the suppression of the disintegration of the base particles. The phrase "not to substantially rotate the disintegration impellers" refers to a state where the disintegration impellers are not rotated at all, or the disintegration impellers are rotated within a range such that the base particles do not undergo disintegration, in consideration of shapes, sizes, and the like of the disintegration impellers, for the purpose of preventing the retention of various raw materials near the disintegration impellers. Concretely, in a case where the disintegration impellers are continuously rotated, the Froude number is preferably 200 or less, and more preferably 100 or less, and in a case where the disintegration impellers are intermittently rotated, the Froude number is not particularly limited. The mixture can be obtained without substantially undergoing disintegration of the base particles by mixing under the conditions as described above.

[0044] The phrase "the base particles substantially maintain their shapes, i.e. the base particles do not undergo disintegration" as used herein refers to a state in which 70% by number or more of the base particles in the mixture maintain their shapes. A method for confirmation thereof includes, for example, a method of observing particles obtained after extracting a soluble component from the resulting mixture with an organic solvent.

[0045] In addition, the Froude number as defined in the present specification is calculated by the following formula:

$$\text{Froude number} = V^2 / (R \times g)$$

wherein V is a peripheral speed [m/s] of a tip end portion of an agitation impeller or disintegration impeller;

R is a rotational radius [m] of an agitation impeller or disintegration impeller; and

g is a gravitational acceleration [m/s²].

[0046] In step B), a powdery raw material other than the base particles can be formulated as desired. The amount of the powdery raw material is preferably 30 parts by weight or less, based on 100 parts by weight of the base particles, from the viewpoint of dissolubility.

[0047] The term "powdery raw material other than the base particles" as used herein means a detergency-fortifying agent or an oil-absorbing agent which is in the form of powder at an ambient temperature. Concretely, the powdery raw materials include base material agents exhibiting a metal ion capturing ability such as zeolite and citrates; base material agents exhibiting an alkalizing ability such as sodium carbonate and potassium carbonate; base material agents exhibiting both a metal ion capturing ability and an alkalizing ability such as crystalline silicates; amorphous silica and amorphous aluminosilicates exhibiting low metal ion capturing ability but high oil-absorbing ability, and the like. By using the above powdery raw material in combination with the base particles as desired, the amount of the surfactant composition formulated can be increased and the deposition of the mixture within the mixer can be reduced, and an improvement in detergency can also be achieved.

[0048] The detergent particles produced according to the present invention may contain c) a nonionic surfactant having a melting point of 30°C or less. In that case, the component c) is added to the base particles in step B). It is preferable that the component c) is added prior to the surfactant composition prepared in step A), to control the structure of liquid crystals and/or crystals in the surfactant composition, thereby increasing the effect of suppressing the bleed-out of the component c).

[0049] The component c) has a melting point of 30°C or less, preferably 25°C or less, and more preferably 22°C or less. As for the component c), for example, a polyoxyethylene-polyoxypropylene block polymer such as a polyoxyalkylene alkyl ether, a polyoxyalkylene alkyl phenyl ether, an alkyl(polyoxyalkylene)polyglycoside, a polyoxyalkylene sorbitan fatty acid ester, a polyoxyalkylene glycol fatty acid ester, a polyoxyethylene-polyoxypropylene-polyoxyethylene alkyl ether (hereinafter abbreviated as EPE nonionic), or a polyoxyalkylene alkylol(fatty acid)amide is preferable.

[0050] Among them, a polyoxyalkylene alkyl ether in which an alkylene oxide is added in an amount of 4 to 12 moles (preferably 6 to 10 moles) to an alcohol having 10 to 14 carbon atoms is preferable. Here, an alkylene oxide includes ethylene oxide, propylene oxide, or the like, and is preferably ethylene oxide.

[0051] In addition, a compound in which ethylene oxide and propylene oxide, and further optionally ethylene oxide, are subjected to a block polymerization or a random polymerization to the above alcohol is preferable, from the viewpoint of dissolubility, especially dissolubility in a low temperature. Among them, the EPE nonionic is preferable.

[0052] These component c) may be used alone or in admixture of two or more kinds. In addition, the nonionic surfactant may be used in the form of an aqueous solution.

[0053] Here, a melting point of the component c) is determined with Mettler FP81 or FP800 Thermo System (manufactured by Mettler Instrumente AG) at a heating rate of 0.2°C/min.

[0054] The component c) is contained in an amount within the range of preferably from 1 to 20% by weight, and more preferably from 5 to 15% by weight of the uni-core detergent particles, from the viewpoint of an improvement in the detergency, an improvement in the anti-caking ability, and the suppression of choking upon becoming powdery.

[0055] In addition, when the uni-core detergent particles produced according to the present invention contain the component c), the component c) may contain, for example, salts of fatty acids, polyethylene glycols, or the like (a molecular weight of from 3,000 to 30,000) as disclosed in JP-B-3161710, to prevent the generation of the bleed-out of the component c) and deterioration of the anti-caking ability. These components are formulated in an amount of preferably from 2 to 40 parts by weight, and more preferably from 2 to 30 parts by weight, based on 100 parts by weight of the component c).

[0056] On the other hand, in the present invention, water contained in the surfactant composition is taken away by the water-soluble inorganic salt, and free-flowability of the surfactant composition is lost, thereby allowing a suppression of the bleed-out of the component c) and an improvement in the anti-caking ability even if the component c) does not contain the above salts of fatty acids, polyethylene glycols, or the like. However, the above salts of fatty acids, polyethylene glycols, or the like may be contained in order to make the suppression of the bleed-out of the component c) and an improvement in the anti-caking ability more effective.

[0057] In addition, as other surfactants, a surfactant which is known in the field of laundry detergents may be added. When an acid precursor such as a linear alkylbenzenesulfonic acid is added, a method of adding an acid precursor such as a linear alkylbenzenesulfonic acid prior to the surfactant composition is preferable in order to suppress the disintegration of the surfactant composition.

[0058] After mixing the surfactant composition or other surfactant, with the base particles, it is preferable that polyethylene glycol (PEG) and/or a fatty acid, and/or soap water is added in an amount of from 1 to 10 parts by weight, based on 100 parts by weight of the base particles to coat the surface of the base particles because the coating improves the anti-caking ability. Further, the addition of PEG and/or a fatty acid and/or soap water is preferable because the addition allows suppression of the aggregation and an increase in dispersibility, thereby improving dissolubility, upon dissolving the detergent particles.

[0059] In addition, the temperature within a mixer during the mixing is preferably a temperature that allows to efficiently mix the surfactant composition and the base particles while substantially suppressing the disintegration of the base particles. For example, a temperature equal to or higher than a pour point of the surfactant composition to be mixed is preferable, more preferably a temperature higher than the pour point by 10°C or more, and especially preferably a temperature higher than the pour point by 20°C or more. In addition, the mixing time is preferably from 2 to 10 minutes or so. The temperature control within the mixer can be carried out by allowing cold water or warm water to flow through a jacket or the like. Therefore, the mixer usable for mixing is preferably a mixer having a construction equipped with a jacket.

[0060] A method for mixing the surfactant composition and the base particles may be a batch process or a continuous process. In the case where mixing is carried out in a batch process, it is preferable that the base particles are previously supplied to a mixer, and thereafter the surfactant composition is added thereto. The temperature at which the surfactant composition is fed is preferably 70°C or less, and more preferably 60°C or less, from the viewpoint of the stability of the surfactant composition.

[0061] In the case where mixing is carried out in a batch process, the mixer is not particularly limited, as long as a mixer which is generally usable for mixing in a batch process is used. For example, as a mixer of which mixing impellers have a paddle shape, (1) a mixer in which blending of powders is carried out by having an agitating shaft in the inner portion of a mixing vessel and attaching agitating impellers on the agitating shaft: for example, Henschel Mixer (manufactured by Mitsui Miike Machinery Co., Ltd.), High-Speed Mixer (manufactured by Fukae Powtec Corp.), Vertical Granulator (manufactured by Powrex Corp.), Lödige Mixer (manufactured by MATSUBO CORPORATION), PLOUGH SHARE Mixer (manufactured by PACIFIC MACHINERY & ENGINEERING Co., LTD.), TSK-MTI Mixer (manufactured by Tsukishima Kikai CO., LTD.) and a mixing machine described in JP-A-Hei-10-296064 or JP-A-Hei-10-296065, or the like; as a mixer of which mixing impellers have a shape of a ribbon-type, (2) a mixer in which blending is carried out by rotating spiral ribbon impellers in a non-rotatable vessel which is cylindrical, semicylindrical, or conical: for example, Ribbon Mixer (manufactured by Nichiwa Kikai Kogyo K.K.), Batch Kneader (manufactured by Satake Kagaku Kikai Kogyo K.K.), Conical Ribbon Mixers/Driers (manufactured by Okawara MFG. CO., LTD.), Julia Mixer (manufactured by TOKUJU CORPORATION), or the like; as a mixer of which mixing impellers have a screw shape, (3) a mixer in which blending is carried out by revolving a screw along a conical vessel, with autorotation centering about a rotating shaft arranged parallel to the vessel wall: for example, Nauta Mixer (manufactured by Hosokawa Micron Corp.), SV Mixer (manufactured by Shinko Pantec Co., Ltd.), or the like.

[0062] In addition, in a case where mixing is carried out in a continuous process, the mixer is not particularly limited,

as long as a continuous mixer which is generally used for a continuous mixing is used. For example, the base particles and the surfactant composition may be mixed by using a continuous-type mixer among the above-mentioned mixers.

[Step C)]

[0063] Step C) is a step of surface-modifying the mixture obtained in step B) with a fine powder. By carrying out this step C), detergent particles having improved free-flowability and anti-caking ability can be obtained.

[0064] As the fine powder, a fine powder of which primary particles have an average particle size of 20 μm or less is preferable, from the viewpoint of improving the coating ratio of the powder particles, and improving free-flowability and anti-caking ability of the powder particles. The average particle size is determined by a method utilizing light scattering, for example a particle analyzer (manufactured by HORIBA, LTD.), or by a microscopic observation.

[0065] As the fine powder, an aluminosilicate is desirable, and an inorganic fine powder such as calcium silicate, silicon dioxide, bentonite, sodium tripolyphosphate, talc, clay, an amorphous silica derivative, or a silicate compound such as a crystalline silicate compound, or a metal soap of which primary particles have a size of 20 μm or less can be used.

[0066] In addition, it is preferable that the fine powder has a high ion exchanging ability and an alkalizing ability, from the viewpoint of detergency.

[0067] The amount of the fine powder used is preferably from 0.5 to 40 parts by weight, and more preferably from 1 to 30 parts by weight, based on 100 parts by weight of the mixture obtained in step B) from the viewpoint of free-flowability and feel of use.

[0068] As the mixing conditions in step C), mixing conditions in which the shape of the base particles containing a surfactant composition is substantially maintained may be selected. Preferred mixing conditions are the use of a mixer equipped with both the agitation impellers and the disintegration impellers. When the mixer as mentioned above is used, the agitation impellers equipped in the mixer have a Froude number of preferably 10 or less, and more preferably 7 or less, from the viewpoint of the suppression of the disintegration of the base particles. The agitation impellers have a Froude number of preferably 2 or more, and even more preferably 3 or more, from the viewpoint of the efficiency of the mixing with the fine powder and the dispersion of the fine powder. Further, the disintegration impellers have a Froude number of preferably 8000 or less, and more preferably 5000 or less, from the viewpoint of the efficiency of mixing with the fine powder and the dispersion of the fine powder. When the Froude number is within this range, uni-core detergent particles having excellent free-flowability can be obtained.

[0069] Preferred mixers include mixers equipped with both the agitation impellers and the disintegration impellers among the mixers usable in step B). In addition, by using separate mixers for step B) and step C), the temperature-control of the mixture is facilitated. For example, when a non-heat resistant component such as perfume or an enzyme is added during the course or after the termination of step C), it is preferable that the mixture is temperature-controlled in step C). The temperature can be controlled by setting a jacket temperature or aeration. In order to efficiently transport the mixture obtained in step B) to the mixer of step C), also a preferred embodiment is to add a part of a fine powder at the termination of step B).

[Uni-Core Detergent Particles]

[0070] Uni-core detergent particles are obtained in the manner as described above. Among them, as the uni-core detergent particles, those containing 20 to 80% by weight of the base particles, 5 to 30% by weight of the component a), a modifying agent fine powder, and separately added detergent components (for example, a fluorescer, an enzyme, a perfume, a defoaming agent, a bleaching agent, a bleaching activator, or the like) are preferable.

[Physical Properties of Uni-Core Detergent Particles]

[0071] In the present invention, the term "uni-core detergent particle" refers to a detergent composition which is produced in which the base particle is used as a core, which is a detergent particle in which a single detergent particle substantially has one base particle as a core.

[0072] As an index for expressing uni-core property of the detergent particles, the degree of particle growth defined by the following formula can be used. The uni-core detergent particles as referred to herein have a degree of particle growth of 1.5 or less, preferably 1.4 or less, and more preferably 1.3 or less. Although its lower limit is not particularly limited, a degree of particle growth of 1.0 or more is preferable.

$$\text{Degree of Particle Growth} = \frac{\text{Average Particle Size of Detergent Particles Obtainable in Step C)}}{\text{Average Particle Size of Base Particles}}$$

[0073] In the above uni-core detergent particle, since the intraparticle aggregation is suppressed, there are some advantages that particles (aggregated particle) having sizes outside the desired particle size range are less likely to be formed, and that their particle size distribution is sharp.

[0074] The uni-core detergent particles have an average particle size of 150 μm or more, preferably from 150 to 500 μm , and more preferably from 180 to 350 μm .

[0075] The uni-core detergent particles have a bulk density of preferably from 300 to 1000 g/L, more preferably from 500 to 1000 g/L, even more preferably from 600 to 1000 g/L, and especially preferably from 650 to 850 g/L.

[0076] In a case where a bulk density is made low as desired in the present invention, a method including the step of, for example, adding a surfactant or the like to a spray-dried slurry, thereby lowering a bulk density of a base particle; formulating a powder raw material having a bulk density lower than a base particle as a powder raw material other than the base particle in step B); reducing an amount of a surfactant composition to be mixed with a base particle; or the like can be employed.

[0077] The uni-core detergent particles have free-flowability, in terms of a flow time, of preferably 10 seconds or shorter, and more preferably 8 seconds or shorter. The flow time refers to a time period required for cascading 100 mL of powder from a hopper used in a measurement of bulk density as defined in JIS K 3362.

[0078] The yield of the uni-core detergent particles is calculated by dividing the weight of a sample passing through a sieve having an opening of 1180 μm by the weight of an entire sample. The yield is preferably 90% or more, and more preferably 95% or more.

[0079] The uni-core detergent particles obtainable by the production method having the constitution as described above have, as mentioned above suppressed particle growth, and sharp particle size distribution, and have improved external appearance and favorable free-flowability, whereby detergent particles having excellent dissolubility can be obtained in a high yield.

[0080] As an index for dissolubility in the present invention, a 60-seconds dissolution ratio of the detergent particles can be used. The dissolution ratio is preferably 80% or more, and more preferably 90% or more.

[0081] The 60-seconds dissolution ratio of the detergent particles is calculated by the method described below.

A 1-L beaker (a cylindrical form having an inner diameter of 105 mm and a height of 150 mm, for instance, a 1-L beaker manufactured by Iwaki Glass Co., Ltd.) is charged with 1 L of hard water cooled to 5°C and having a water hardness corresponding to 71.2 mg CaCO_3/L (a molar ratio of Ca/Mg: 7/3). With keeping the water temperature constant at 5°C with a water bath, water is stirred with a stirring bar [length: 35 mm and diameter: 8 mm, for instance, Model "TEFLON SA" (MARUGATA-HOSOGATA), manufactured by ADVANTEC] at a rotational speed (800 r/min), such that a depth of swirling to the water depth is about 1/3. The detergent particles which are accurately sample-reduced and weighed so as to be 1.0000 g $\pm$ 0.0010 g are supplied and dispersed in water with stirring, and stirring is continued. After 60 seconds from supplying the particles, a liquid dispersion of the detergent particles in the beaker is filtered with a standard sieve (diameter: 100 mm) having a sieve-opening of 74 μm as defined by JIS Z 8801 of a known weight. Thereafter, water-containing detergent particles remaining on the sieve are collected in an open vessel of a known weight together with the sieve. Incidentally, the operation time from the start of filtration to collection of the sieve is set at 10 sec $\pm$ 2 sec. The insoluble remnants of the collected detergent particles are dried for one hour in an electric dryer heated to 105°C. Thereafter, the dried insoluble remnants are cooled by keeping in a desiccator with a silica gel (25°C) for 30 minutes. After cooling the insoluble remnants, a total weight of the dried insoluble remnants of the detergent, the sieve and the collecting vessel is measured, and the dissolution ratio (%) of the detergent particles is calculated by the formula (2):

$$\text{Dissolution Ratio (\%)} = \{1 - (T/S)\} \times 100 \quad (2)$$

wherein S is a weight (g) of the detergent particles supplied; and T is a dry weight (g) of insoluble remnants of the detergent particles remaining on the sieve when an aqueous solution prepared under the above stirring conditions is filtered with the sieve (drying conditions: maintaining at a temperature of 105°C for 1 hour, and thereafter maintaining for 30 minutes in a desiccator (25°C) containing silica gel).

[0082] In addition, the detergent particles of the present invention are excellent in bleed-out preventing property of a nonionic surfactant. The bleed-out property of a nonionic surfactant will be evaluated as follows.

[0083] An open-top box having dimensions of 10.2 cm in length, 6.2 cm in width, and 4 cm in height is made out of a filter paper (No. 2, manufactured by ADVANTEC) by stapling the filter paper at four corners. Two lines are previously drawn with an oil-based magic marker to be crossed with each other, along the diagonals of the portion corresponding the bottom of the box. The box is charged with a 200 mL sample, and sealed in an acrylic casing. The sample is allowed to stand in a thermostat at a temperature of 30°C for 7 days, and the bleed-out property of a nonionic surfactant is evaluated.

[0084] The judgment is made by visually examining the extent of bleeding of the oil-based magic marker drawn on the bottom of the box after discharging the sample. The evaluation was made on 1 to 5 ranks, and the state of each rank is as follows.

Rank 1: not bleeding at all;

Rank 2: bleeding is generated in a part of the lines, like a state in which cilia are grown;

Rank 3: bleeding is generated in almost an entire line, an average thickness of the magic marker line being less than 2.0 times;

Rank 4: bleeding is generated in an entire line, an average thickness of the magic marker line being 2.0 times or more and less than 3.0 times; and

Rank 5: bleeding is generated in an entire line, an average thickness of the magic marker line being 3.0 times or more.

[0085] In the present invention, those of which evaluation of the extent of bleeding mentioned above has a rank of 1 and 2 are acceptable products.

EXAMPLES

[0086] The present invention will be further described hereinbelow by the Examples given below.

Example 1

[0087] Base particles used in Examples 1-1 to 1-8 were produced by the following procedures. The amount 460 kg of water was added to a 1 m³-mixing vessel having agitation impellers. After the water temperature reached 55°C, 120 kg of sodium sulfate, 140 kg of sodium carbonate and 5 kg of sodium sulfite were added thereto. After agitating the mixture for 10 minutes, 170 kg of a 40% by weight-aqueous sodium polyacrylate solution was added thereto. After agitating the mixture for additional 10 minutes, 40 kg of sodium chloride and 140 kg of zeolite were added thereto, and the resulting mixture was agitated for 30 minutes, to obtain a homogeneous slurry. The final temperature of this slurry was 58°C.

[0088] This slurry was sprayed at a spraying pressure of 25 kg/cm² from a pressure spray nozzle arranged near the top of a spray-drying tower. A high-temperature gas to be fed to the spray-drying tower was supplied at a temperature of 225°C to the bottom of the tower and exhausted at a temperature of 105°C from the top of the tower. The water content of the base particles was 1.6%.

[0089] The resulting base particles had physical properties such that the base particles had an average particle size of 281 μm, a bulk density of 506 g/L, a free-flowability of 5.8 seconds, and a supporting ability of 45 mL/100 g.

[0090] Base particles used in Examples 1-9 to 1-10 were produced by the following procedures.

The amount 430 kg of water was added to a 1 m³-mixing vessel having agitation impellers. After the water temperature reached 55°C, 160 kg of sodium sulfate was added thereto. After agitating the mixture for 5 minutes, 100 kg of sodium silicate (effective ingredient: 40%) and 10 kg of carboxymethyl cellulose were added thereto. After agitating the mixture for 5 minutes, 60 kg of sodium tripolyphosphate and 130 kg of sodium carbonate were added thereto. After agitating the mixture for 15 minutes, 60 kg of a 40% by weight-aqueous sodium polyacrylate solution was added thereto. The resulting mixture was agitated for 30 minutes, to obtain a homogeneous slurry. The final temperature of this slurry was 60°C.

[0091] This slurry was sprayed at a spraying pressure of 40 kg/cm² from a pressure spray nozzle arranged near the top of a spray-drying tower. A high-temperature gas to be fed to the spray-drying tower was supplied at a temperature of 235°C to the bottom of the tower and exhausted at a temperature of 115°C from the top of the tower. The water content of the base particles was 2.0%.

[0092] The resulting base particles had physical properties such that the base particles had an average particle size of 203 μm, a bulk density of 420 g/L, a free-flowability of 6.4 seconds, and a supporting ability of 32 mL/100 g.

[0093] Base particles used in Example 1-11 were produced by the following procedures.

The amount 413 kg of water was added to a 1 m³-mixing vessel having agitation impellers. After the water temperature reached 55°C, 135 kg of sodium sulfate was added thereto. After agitating the mixture for 5 minutes, 60 kg of sodium

silicate (effective ingredient: 40%) and 12 kg of carboxymethyl cellulose were added thereto. After agitating the mixture for 5 minutes, 50 kg of sodium tripolyphosphate and 150 kg of sodium carbonate were added thereto. After agitating the mixture for 15 minutes, 130 kg of a 40% by weight-aqueous sodium polyacrylate solution was added thereto. After agitating the resulting mixture for additional 10 minutes, 50 kg of sodium chloride was added thereto, and the resulting mixture was agitated for 30 minutes, to obtain a homogeneous slurry. The final temperature of this slurry was 60°C.

[0094] This slurry was sprayed at a spraying pressure of 35 kg/cm² from a pressure spray nozzle arranged near the top of a spray-drying tower. A high-temperature gas to be fed to the spray-drying tower was supplied at a temperature of 235°C to the bottom of the tower and exhausted at a temperature of 112°C from the top of the tower. The water content of the base particles was 1.2%.

[0095] The resulting base particles had physical properties such that the base particles had an average particle size of 240 μm, a bulk density of 374 g/L, a free-flowability of 6.0 seconds, and a supporting ability of 30 mL/100 g.

[0096] In addition, the components of the surfactant composition used in Examples 1-1 to 1-11 and Comparative Examples 1-1 to 1-2 are those as listed in Table 1.

Example 1-1

[0097] One-hundred parts by weight of the base particles previously heated to 50°C and powder raw materials in amounts of parts by weight listed in Table 2 were supplied into Lödige Mixer (manufactured by MATSUBO CORPORATION; capacity: 20 L, equipped with a jacket), and the rotation of a main shaft was started (rotational speed of main shaft: 80 r/min, Froude number of agitation impellers: 1.07). Here, hot water at 80°C was allowed to flow through the jacket at 10 L/minute, without rotating a chopper (equipped with disintegration impellers). After agitating the components with the rotation of the main shaft for 1 minute, 44 parts by weight of a surfactant composition at 60°C was supplied over 2 minutes, and the components were then mixed for 6 minutes. The rotations were temporarily stopped, and 5.3 parts by weight of a crystalline silicate listed in Table 2 was supplied into the mixer. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 15 seconds. The rotation was temporarily stopped, and 13 parts by weight of a fine powder (zeolite) was supplied thereto. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 30 seconds, and the resulting detergent particles were then discharged. The physical properties of the resulting detergent particles were as listed in Table 2.

[0098] Incidentally, in Table 2, in addition to an average particle size (entire particles) of the detergent particles, an average particle size of the detergent particles that passed through the sieve having an opening of 1180 μm used in the calculation of yield was also listed together. The free-flowability, the bulk density, and the dissolution ratio of the detergent particles, and the bleed-out property of the component c) were determined and/or evaluated using detergent particles which were allowed to pass through the above-mentioned sieve to exclude aggregated or coarse particles.

[0099]

55 50 45 40 35 30 25 20 15 10 5

[Table 1]

		Ex.											Comp. Ex.	
		1-1	1-2	1-3	1-4	1-5	1-6	1-7	1-8	1-9	1-10	1-11	1-1	1-2
Surfactant Composition * 1														
	a) R-OSO ₃ Na(C12/14/16=67/27/6)	100	-	100	100	100	100	100	100	100	100	-	100	100
	a) R-OSO ₃ Na (C14=100)	-	100	-	-	-	-	-	-	-	-	100	-	-
	b) Water	38.9	42.9	38.9	38.9	38.9	38.9	38.9	38.9	39	39	42.9	38.9	38.9
Viscosity [Pa·s] of Surfactant Composition at 60°C		4.2	3.7	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	3.7 -	4.2	4.2
	*1: parts by weight.													

[0100]

5

10

15

20

25

30

35

40

45

50

55

[Table 2]

Composition of Detergent Particles (parts by weight)			Ex.										Comp. Ex				
			1-1	1-2	1-3	1-4	1-5	1-6	1-7	1-8	1-9	1-10	1-11	1-1	1-2		
	<u>Surfactant Composition</u>		44	45	31	31	31	47	15	31	31	22	34	44	44		
	c) Polyoxyethylene Alkyl Ether		-	-	22	22	22	11	32	22	0	16		-	-		
	Polyethylene Glycol		-	-	-	-	-	-	-	-	-	1.5	1	-	-		
	Fatty Acid		-	-	-	-	-	-	-	-	-	-		-	-		
	Base Particles		100	100	100	100	100	100	100	100	100	100	100	-	-		
	Base Particle Substitute Powder		-	-	-	-	-	-	-	-	-			100	100		
	Raw Material Powder																
		Sodium Carbonate	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2		3.2	3.2	
		Crystalline Silicate	14	14	14	14	14	14	14	14	14	14	14		14	14	
	<u>Fine Powder</u>																
			Crystalline Silicate	5.3	5.3	5.3	5.3	5.3	5.3	5.3	5.3	5.3	5.3		5.3	5.3	
			Zeolite	13	13	13	13	13	13	13	13	13	13	0	34	85	64
			Sodium Tripolyphosphate	-	-	-	-	-	-	-	-	-	-	20		-	-
Average Particle Size of Detergent Particles [μm] (entire particles)			351	363	334	311	326	329	322	345	246	223	265	898	886		
Yield [%]			91	91	97	98.6	97	93	99	97	94	99	98	57	58		
Degree of Particle Growth [-]			1.249	1.2918	1.19	1.1068	11.643	11.7	11.5	1.2278	1.21	1.1	1.1	-	-		
Average Particle Size of Detergent Particles [μm] (those having sizes of 1180 μm-sieve passed)			312	318	306	308	303	304	318	322	229	220	264	591	553		
Free-Flowability of Detergent Particles [s]			6.1	6.3	6.6	6.6	6.5	6.7	6.7	6.5	6.9	6.8	7	7.3	8.6		
Bulk Density of Detergent Particles [g/L]			580	612	644	677	652	641	681	643	551	585	468	815	862		

(continued)

Composition of Detergent Particles (parts by weight)	Ex.											Comp. Ex	
	1-1	1-2	1-3	1-4	1-5	1-6	1-7	1-8	1-9	1-10	1-11	1-1	1-2
Dissolution Ratio of Detergent Particles [%]	81.1	81	96	97	94	91	99	95	84	98	99	61.3	62.6
Bleed-out Property of Component c)	-	-	1	2	2	1	2	1	-	1	-	-	-

[0101] In Tables 1 and 2, the followings were used.

Sodium Carbonate: manufactured by Central Glass Co., Ltd. under the trade name of DENSE ASH, average particle size: 290 μm , bulk density: 980 g/L;

Crystalline Silicate: manufactured by K.K. Tokuyama Siltex under the trade name of Prefeed N (a powder pulverized to a size of an average particle size of 18 μm);

Zeolite: manufactured by Zeobuilder under the trade name of Zeobuilder (zeolite 4A-type, average particle size 3.5 μm);

Sodium tripolyphosphate: manufactured by SHIMONOSEKI MITSUI CHEMICALS, INC. under the trade name of sodium tripolyphosphate (a powder pulverized to size of an average particle size of 15 μm);

Polyoxyethylene Alkyl Ether: manufactured by Kao Corporation under the trade name of EMULGEN 108KM (average number of moles of ethylene oxide added: 8.5, number of carbon atoms of alkyl moiety: 12-14), melting point: 18°C);

Polyethylene Glycol: manufactured by Kao Corporation under the trade name of K-PEG6000LA (average molecular weight: 8500, melting point: 60°C); and

Fatty Acid: manufactured by Kao Corporation under the trade name of LUNAC P-95.

[0102] The component b) in the surfactant composition listed in Table 1 was 39 parts by weight, based on 100 parts by weight of the component a), and the viscosity of the surfactant composition was 4.2 Pa·s (60°C).

Example 1-2

[0103] Detergent particles were obtained in the same manner as in Example 1-1 with the components listed in Table 2. The physical properties of the resulting detergent particles are shown in Table 2.

[0104] Here, as the component a) in the surfactant composition used in Example 1-2, one having an alkyl chain length of 14 was used. The components and viscosity are as shown in Table 1.

Example 1-3

[0105] One-hundred parts by weight of the base particles previously heated to 50°C and powder raw materials in amounts of parts by weight listed in Table 2 were supplied into Lödige Mixer (manufactured by MATSUBO CORPORATION; capacity: 20 L, equipped with a jacket), and the rotation of a main shaft was started (rotational speed of main shaft: 80 r/min, Froude number of agitation impellers: 1.07). Here, hot water at 80°C was allowed to flow through the jacket at 10 L/minute, without rotating a chopper (equipped with disintegration impellers). After agitating with the rotation of the main shaft for 1 minute, 22 parts by weight of the polyoxyethylene alkyl ether at 60°C was supplied over 1 minute, and subsequently 31 parts by weight of a surfactant composition at 60°C was supplied over 1 minute, and the components were then mixed for 6 minutes. The rotations were temporarily stopped, and 5.3 parts by weight of a crystalline silicate listed in Table 2 was supplied into the mixer. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 15 seconds. The rotation was temporarily stopped, and 13 parts by weight of a fine powder (zeolite) was supplied thereto. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 30 seconds, and the resulting detergent particles were discharged. The physical properties of the resulting detergent particles were as listed in Table 2.

[0106] Here, in Example 1-3, as the surfactant composition, the same one as that used in Example 1-1 was used. The components and viscosity are as shown in Table 1.

Example 1-4

[0107] Detergent particles were obtained in the same manner as in Example 1-3 with the components listed in Table 2, provided that the polyoxyethylene alkyl ether and the surfactant composition were previously mixed and then added over 2 minutes. The physical properties of the resulting detergent particles are shown in Table 2.

[0108] Here, the surfactant composition used in Example 1-4 was the same one as that used in Example 1-1. The components and viscosity are as shown in Table 1.

Example 1-5

[0109] Detergent particles were obtained in the same manner as in Example 1-3 with the components listed in Table 2, except that the surfactant composition was supplied over 1 minute, and thereafter the polyoxyethylene alkyl ether was supplied over 1 minute. The physical properties of the resulting detergent particles are shown in Table 2.

[0110] Here, the surfactant composition used in Example 1-5 was the same one as that used in Example 1-1. The components and viscosity are as shown in Table 1.

Examples 1-6 and 1-7

[0111] Detergent particles were obtained in the same manner as in Example 1-3 with the components listed in Table 2, except for the amounts of the polyoxyethylene alkyl ether and the surfactant composition. The physical properties of the resulting detergent particles are shown in Table 2.

[0112] Here, the surfactant compositions used in Examples 1-6 and 1-7 were the same one as that used in Example 1-1. The components and viscosity are as shown in Table 1.

Example 1-8

[0113] The polyoxyethylene alkyl ether was supplied and the surfactant composition was then supplied in the same manner as in Example 1-3 with the components listed in Table 2, provided that 2.0 parts by weight of polyethylene glycol was previously mixed with the polyoxyethylene alkyl ether, and the mixture was then added. After mixing the components for 4 minutes, 3.6 parts by weight of the fatty acid was added thereto over 1 minute, subsequently mixing was carried out for 1 minute, and the rotations were temporarily stopped. The subsequent procedures were carried out in the same manner as in Example 1-3. The physical properties of the resulting detergent particles are shown in Table 2.

[0114] Here, the surfactant composition used in Example 1-8 was the same one as that used in Example 1-1. The components and viscosity are as shown in Table 1.

[0115] It can be seen that in all of Examples 1-3 to 1-8 in which the component c) the polyoxyethylene alkyl ether was added the bleed-out of the component c) is suppressed. Among them, the bleed-out can be further suppressed by mixing the component c) with the base particles prior to mixing with the surfactant composition. In addition, it can be seen that similar effects are caused by mixing the polyethylene glycol with the component c). In addition, the detergent particles to which the component c) was added did not give a feel of choking upon handling.

Example 1-9

[0116] Detergent particles were obtained in the same manner as in Example 1-1 with the components listed in Table 2. The physical properties of the resulting detergent particles are shown in Table 2.

Example 1-10

[0117] Detergent particles were obtained in the same manner as in Example 1-3 with the components listed in Table 2. Here, as a fine powder, sodium tripolyphosphate was used. The physical properties of the resulting detergent particles are shown in Table 2.

Example 1-11

[0118] The powder raw material composed of 100 parts by weight of the base particles previously heated to 50°C was supplied into Lödige Mixer (manufactured by MATSUBO CORPORATION; capacity: 20 L, equipped with a jacket), and the rotation of a main shaft was started (rotational speed of main shaft: 80 r/min, Froude number of agitation impellers: 1.07). Incidentally, hot water at 80°C was allowed to flow through the jacket at 10 L/minute, without rotating a chopper (equipped with disintegration impellers). After agitating the components with the rotation of the main shaft for 1 minute, 1.0 part by weight of the polyethylene glycol at 60°C was supplied over 1 minute, and subsequently 34 parts by weight of a surfactant composition at 60°C was supplied over 2 minutes, and the components were then mixed for 6 minutes. The rotations were temporarily stopped, and 34 parts by weight of a fine powder (zeolite) was supplied thereto. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 30 seconds, and the resulting detergent particles were discharged. The physical properties of the resulting detergent particles were as listed in Table 2.

[0119] Incidentally, in Table 2, in addition to an average particle size (entire particles) of the detergent particles, an average particle size, of the detergent particles that passed through the sieve having an opening of 1180 μm used in the calculation of yield was also listed together. The free-flowability, the bulk density, and the dissolution ratio of the detergent particles, and the bleed-out property of the component c) were determined and/or evaluated using detergent particles which were allowed to pass through the above-mentioned sieve to exclude aggregated or coarse particles.

Comparative Example 1-1

[0120] Detergent particles were obtained in the same manner as in Example 1-1 with the components listed in Table 2, using a base particle substitute powder in place of the base particles. Here, in Comparative Example 1-1, as the base particle substitute powder, a powder produced by dry-blending the components so as to have the ratio of the powder raw material blended in the base particles in a given compositional ratio was used. The physical properties of the resulting detergent particles are shown in Table 2. Detergent particles having excellent free-flowability were obtained in the same manner as in Examples 1-1 to 1-11; however, the amount of a modifying agent fine powder (zeolite) which was necessary to obtain a detergent having excellent free-flowability was dramatically increased as compared to those of Examples 1-1 to 1-11. Also, the aggregation and formation of coarse particles of the particles took place, thereby dramatically lowering its yield. In addition, its dissolution ratio was lowered. The amount and yield of the fine powder added at this time, and the average particle size, the free-flowability, the bulk density, and the dissolution ratio of the detergent particles are shown in Table 2.

[0121] Here, the surfactant composition used in Comparative Example 1-1 was the same one as that used in Example 1-1. The components and water content and viscosity are as shown in Table 1.

Comparative Example 1-2

[0122] Detergent particles were obtained in the same manner as in Example 1-1 with the components listed in Table 2, using a base particle substitute powder in the same manner as in Comparative Example 1. Here, in Comparative Example 2, as the base particle substitute powder, a powder produced by dry-blending sodium bicarbonate and LIGHT ASH in a ratio of sodium bicarbonate / LIGHT ASH = 2/1 was used. The physical properties of the resulting detergent particles are shown in Table 2.

[0123] The amount of a modifying agent fine powder (zeolite) which was necessary to improve its free-flowability was dramatically increased as compared to those of Examples 1-1 to 1-11. Also, the aggregation and formation of coarse particles of the particles took place, thereby dramatically lowering its yield.

[0124] The amount and yield of the fine powder (zeolite) added at this time, and the average particle size, the free-flowability, the bulk density, and the dissolution ratio of the detergent particles are shown in Table 2.

[0125] Here, the surfactant composition used in Comparative Example 1-2 was the same one as that used in Example 1-1.

[0126] It can be seen from the results of Table 2 that all of the detergent particles obtained in Examples 1-1 to 1-11 are excellent in free-flowability, dissolution ratio, and yield, as compared to those of Comparative Examples 1-1 and 1-2.

Example 2

[0127] Base particles used in Examples 2-1 to 2-6 were produced by the following procedures. The amount 460 kg of water was added to a 1 m^3 -mixing vessel having agitation impellers. After the water temperature reached 55°C, 120 kg of sodium sulfate, 140 kg of sodium carbonate and 5 kg of sodium sulfite were added thereto. After agitating the mixture for 10 minutes, 170 kg of a 40% by weight-aqueous sodium polyacrylate solution was added thereto. After agitating the mixture for additional 10 minutes, 40 kg of sodium chloride and 140 kg of zeolite were added thereto, and the resulting mixture was agitated for 30 minutes, to obtain a homogeneous slurry. The final temperature of this slurry was 58°C.

[0128] This slurry was sprayed at a spraying pressure of 25 kg/cm^2 from a pressure spray nozzle arranged near the top of a spray-drying tower. A high-temperature gas to be fed to the spray-drying tower was supplied at a temperature of 225°C to the bottom of the tower and exhausted at a temperature of 105°C from the top of the tower. The water content of the base particles was 1.6%.

[0129] The resulting base particles had physical properties such that the base particles had an average particle size of 281 μm , a bulk density of 506 g/L, a free-flowability of 5.8 seconds, and a supporting ability of 45 mL/100 g.

[0130] Base particles used in Examples 2-7 to 2-8 were produced by the following procedures. The amount 430 kg of water was added to a 1 m^3 -mixing vessel having agitation impellers. After the water temperature reached 55°C, 160 kg of sodium sulfate was added thereto. After agitating the mixture for 5 minutes, 100 kg of sodium silicate (effective ingredient: 40%) and 10 kg of carboxymethyl cellulose were added thereto. After agitating the mixture

for 5 minutes, 60 kg of sodium tripolyphosphate and 130 kg of sodium carbonate were added thereto. After agitating the mixture for 15 minutes, 60 kg of a 40% by weight-aqueous sodium polyacrylate solution was added thereto. The resulting mixture was agitated for 30 minutes, to obtain a homogeneous slurry. The final temperature of this slurry was 60°C.

[0131] This slurry was sprayed at a spraying pressure of 40 kg/cm² from a pressure spray nozzle arranged near the top of a spray-drying tower. A high-temperature gas to be fed to the spray-drying tower was supplied at a temperature of 235°C to the bottom of the tower and exhausted at a temperature of 115°C from the top of the tower. The water content of the base particles was 2.0%.

[0132] The resulting base particles had physical properties such that the base particles had an average particle size of 203 µm, a bulk density of 420 g/L, a free-flowability of 6.4 seconds, and a supporting ability of 32 mL/100 g.

[0133] Base particles used in Example 2-9 were produced by the following procedures.

The amount 413 kg of water was added to a 1 m³-mixing vessel having agitation impellers. After the water temperature reached 55 °C, 135 kg of sodium sulfate was added thereto. After agitating the mixture for 5 minutes, 60 kg of sodium silicate (effective ingredient: 40%) and 12 kg of carboxymethyl cellulose were added thereto. After agitating the mixture for 5 minutes, 50 kg of sodium tripolyphosphate and 150 kg of sodium carbonate were added thereto. After agitating the mixture for 15 minutes, 130 kg of a 40% by weight-aqueous sodium polyacrylate solution was added thereto. After agitating the resulting mixture for additional 10 minutes, 50 kg of sodium chloride was added thereto, and the resulting mixture was agitated for 30 minutes, to obtain a homogeneous slurry. The final temperature of this slurry was 60°C.

[0134] This slurry was sprayed at a spraying pressure of 35 kg/cm² from a pressure spray nozzle arranged near the top of a spray-drying tower. A high-temperature gas to be fed to the spray-drying tower was supplied at a temperature of 235°C to the bottom of the tower and exhausted at a temperature of 112°C from the top of the tower. The water content of the base particles was 1.2%.

[0135] The resulting base particles had physical properties such that the base particles had an average particle size of 240 µm, a bulk density of 374 g/L, a free-flowability of 6.0 seconds, and a supporting ability of 30 mL/100 g.

[0136] In addition, the components of the surfactant composition used in Examples 2-1 to 2-9 and Comparative Examples 2-1 to 2-2 are those as listed in Table 3.

Example 2-1

[0137] One-hundred parts by weight of the base particles previously heated to 50°C and powder raw materials in amounts of parts by weight listed in Table 4 were supplied into Lödige Mixer (manufactured by MATSUBO CORPORATION; capacity: 20 L, equipped with a jacket), and the rotation of a main shaft was started (rotational speed of main shaft: 80 r/min, Froude number of agitation impellers: 1.07). Here, hot water at 80°C was allowed to flow through the jacket at 10 L/minute, without rotating a chopper (equipped with disintegration impellers). After agitating the components with the rotation of the main shaft for 1 minute, 64 parts by weight of a surfactant composition at 60°C was supplied over 2 minutes, and the components were then mixed for 6 minutes. The rotations were temporarily stopped, and 5.3 parts by weight of a crystalline silicate listed in Table 4 was supplied into the mixer. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 15 seconds. The rotation was temporarily stopped, and 42 parts by weight of a fine powder (zeolite) was supplied thereto. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 30 seconds, and the resulting detergent particles were then discharged. The physical properties of the resulting detergent particles were as listed in Table 4.

[0138] Incidentally, in Table 4, in addition to an average particle size (entire particles) of the detergent particles, an average particle size of the detergent particles that passed through the sieve having an opening of 1180 µm used in the calculation of yield was also listed together. The free-flowability, the bulk density, and the dissolution ratio of the detergent particles, and the bleed-out property of the component c) were determined and/or evaluated using detergent particles which were allowed to pass through the above-mentioned sieve to exclude aggregated or coarse particles.

[0139]

[Table 3]

	Ex.									Comp. Ex.	
	2-1	2-2	2-3	2-4	2-5	2-6	2-7	2-8	2-9	2-1	2-2
Surfactant Composition											

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(continued)

		Ex.									Comp. Ex.	
		2-1	2-2	2-3	2-4	2-5	2-6	2-7	2-8	2-9	2-1	2-2
5	a) R-O-(CH ₂ CH ₂ O) ISO ₃ Na(R:ClZ/ 14=72/28)	100	-	100	100	100	100	100	100	-	100	100
10	a) R- O-(CH ₂ CH ₂ O) ₂ SO ₃ Na	-	100	-	-	-	-	-	-	100	-	-
	b) Water	43	41	43	43	43	43	43	43	41	43	43
15	Viscosity of Surfactant Composition [Pa·s] at 60°C	3.1	2.8	3.2	3.1	3.2	3.1	3.1	3.1	2.8	3.1	3.1

[0140]

[Table 4]

Composition of Detergent Particles (parts by weight)			Ex.								Comp. Ex.			
			2-1	2-2	2-3	2-4	2-5	2-6	2-7	2-8	2-9	2-1	2-2	
	<u>Surfactant Composition</u>		64	63	32	32	32	32	45	26	34	64	64	
	c) Polyoxyethylene Alkyl Ether		-	-	22	22	22	22	-	16	-	-	-	
	Polyethylene Glycol		-	-	-	-	-	2	-	-	1	-	-	
	Fatty Acid		-	-	-	-	-	3.6	-	-	-	-	-	
	Base Particles		100	100	100	100	100	100	100	100	100	-	-	
	Base Particle Substitute Powder		-	-	-	-	-	-	-	-	-	100	100	
	Raw Material Powder													
		Sodium Carbonate		3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2	-	3.2	3.2
		Crystalline Silicate		14	14	14	14	14	14	14	14	-	14	14
	<u>Fine Powder</u>													
			Crystalline Silicate	5.3	5.3	5.3	5.3	5.3	5.3	5.3	5.3	-	5.3	5.3
			Zeolite	42	34	11	11	11	11	40	-	34	85	59
			Sodium Tripolyphosphate	-	-	-	-	-	-	-	13	-	-	-
Average Particle Size of Detergent Particles [μm] (entire particles)			396	377	296	300	303	286	254	228	262	881	927	
Yield [%]			93	99	99	98.6	99	99	96	99	99	64	57.8	
Degree of Particle Growth [-]			1.4	1.3	1.053	1.068	1.078	1.018	1.3	1.1	1.1	-	-	
Average Particle Size of Detergent Particles [μm] (those having sizes of 1180 μm-sieve passed)			319	317	295	300	302	286	253	227	261	535	327	
Free-Flowability of Detergent Particles [s]			6.3	5.6	5.8	7.1	6.4	6.5	6	6.2	6.8	6.6	8.6	
Bulk Density of Detergent Particles [g/L]			747	769	769	741	751	752	740	730	489	923	853	
Dissolution Ratio of Detergent Particles [%] Bleed-out Property of Component c)			94	96	99	99	99	98	98	98	99	70	89	
			-	-	1	2	2	1	-	1	-	-	-	

[0141] In Tables 3 and 4, the followings were used.

Sodium Carbonate: manufactured by Central Glass Co., Ltd. under the trade name of DENSE ASH, average particle size: 290 μm , bulk density: 980 g/L;

Crystalline Silicate: manufactured by K.K. Tokuyama Siltex under the trade name of Prefeed N (a powder pulverized to a size of an average particle size of 18 μm);

Zeolite: manufactured by Zeobuilder under the trade name of Zeobuilder (zeolite 4A-type, average particle size 3.5 μm);

Sodium tripolyphosphate: manufactured by SHIMONOSEKI MITSUI CHEMICALS, INC. under the trade name of sodium tripolyphosphate (a powder pulverized to size of an average particle size of 15 μm);

Polyoxyethylene Alkyl Ether: manufactured by Kao Corporation under the trade name of EMULGEN 108KM (average number of moles of ethylene oxide added: 8.5, number of carbon atoms of alkyl moiety: 12-14), melting point: 18°C);

Polyethylene Glycol: manufactured by Kao Corporation under the trade name of K-PEG6000LA (average molecular weight: 8500, melting point: 60°C); and

Fatty Acid: manufactured by Kao Corporation under the trade name of LUNAC P-95.

[0142] The component b) in the surfactant composition listed in Table 3 was 43 parts by weight, based on 100 parts by weight of the component a), and the viscosity of the surfactant composition was 3.1 Pa·s (60°C).

Example 2-2

[0143] Detergent particles were obtained in the same manner as in Example 2-1 with the components listed in Table 4. The physical properties of the resulting detergent particles are shown in Table 4.

[0144] Here, the surfactant composition used in Example 2-2 is a product commercially available under the trade name of EMAL270J (average number of moles of EO = 2) (manufactured by Kao Corporation), and its components and viscosity are as shown in Table 1.

Example 2-3

[0145] One-hundred parts by weight of the base particles previously heated to 50°C and powder raw materials in amounts of parts by weight listed in Table 4 were supplied into Lödige Mixer (manufactured by MATSUBO CORPORATION; capacity: 20 L, equipped with a jacket), and the rotation of a main shaft was started (rotational speed of main shaft: 80 r/min, Froude number of agitation impellers: 1.07). Here, hot water at 80°C was allowed to flow through the jacket at 10 L/minute, without rotating a chopper (equipped with disintegration impellers). After agitating with the rotation of the main shaft for 1 minute, 22 parts by weight of the polyoxyethylene alkyl ether at 60°C was supplied over 1 minute, and subsequently 32 parts by weight of a surfactant composition at 60°C was supplied over 1 minute, and the components were then mixed for 6 minutes. The rotations were temporarily stopped, and 5.3 parts by weight of a crystalline silicate listed in Table 2 was supplied into the mixer. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 15 seconds. The rotation was temporarily stopped, and 11 parts by weight of a fine powder (zeolite) was supplied thereto. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 30 seconds, and the resulting detergent particles were discharged. The physical properties of the resulting detergent particles were as listed in Table 4.

[0146] Here, in Example 2-3, as the surfactant composition, the same one as that used in Example 2-1 was used. The components and viscosity are as shown in Table 3.

Example 2-4

[0147] Detergent particles were obtained in the same manner as in Example 2-3 with the components listed in Table 4, provided that the polyoxyethylene alkyl ether and the surfactant composition were previously mixed and then added over 2 minutes. The physical properties of the resulting detergent particles are shown in Table 4.

[0148] Here, the surfactant composition used in Example 2-4 was the same one as that used in Example 2-1. The components and viscosity are as shown in Table 3.

Example 2-5

[0149] Detergent particles were obtained in the same manner as in Example 2-3 with the components listed in Table 4, except that the surfactant composition was supplied over 1 minute, and thereafter the polyoxyethylene alkyl ether was supplied over 1 minute. The physical properties of the resulting detergent particles are shown in Table 4.

[0150] Here, the surfactant composition used in Example 2-5 was the same one as that used in Example 2-1. The components and viscosity are as shown in Table 3.

Example 2-6

[0151] The polyoxyethylene alkyl ether was supplied and the surfactant composition was then supplied in the same manner as in Example 2-3 with the components listed in Table 4, provided that 2.0 parts by weight of polyethylene glycol was previously mixed with the polyoxyethylene alkyl ether, and the mixture was then added. The physical properties of the resulting detergent particles are shown in Table 4. After mixing the components for 4 minutes, 3.6 parts by weight of the fatty acid was added thereto over 1 minute, subsequently mixing was carried out for 1 minute, and the rotations were temporarily stopped. The subsequent procedures were carried out in the same manner as in Example 2-3.

[0152] Here, the surfactant composition used in Example 2-6 was the same one as that used in Example 2-1. The components and viscosity are as shown in Table 3.

[0153] It can be seen that in all of Examples 2-3 to 2-6 in which the component c) the polyoxyethylene alkyl ether was added the bleed-out of the component c) is suppressed. Among them, the bleed-out can be further suppressed by mixing the component c) with the base particles prior to mixing with the surfactant composition. In addition, it can be seen that similar effects are caused by mixing the polyethylene glycol with the component c).

Example 2-7

[0154] Detergent particles were obtained in the same manner as in Example 2-1 with the components listed in Table 4. The physical properties of the resulting detergent particles are shown in Table 4.

Example 2-8

[0155] Detergent particles were obtained in the same manner as in Example 2-3 with the components listed in Table 4. Here, as a fine powder, sodium tripolyphosphate was used. The physical properties of the resulting detergent particles are shown in Table 4.

Example 2-9

[0156] The powder raw material composed of 100 parts by weight of the base particles previously heated to 50°C was supplied into Lödige Mixer (manufactured by MATSUBO CORPORATION; capacity: 20 L, equipped with a jacket), and the rotation of a main shaft was started (rotational speed of main shaft: 80 r/min, Froude number of agitation impellers: 1.07). Incidentally, hot water at 80°C was allowed to flow through the jacket at 10 L/minute, without rotating a chopper (equipped with disintegration impellers). After agitating the components with the rotation of the main shaft for 1 minute, 1.0 part by weight of the polyethylene glycol at 60°C was supplied over 1 minute, and subsequently 34 parts by weight of a surfactant composition at 60°C was supplied over 2 minutes, and the components were then mixed for 6 minutes. The rotations were temporarily stopped, and 34 parts by weight of a fine powder (zeolite) was supplied thereto. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 30 seconds, and the resulting detergent particles were discharged. The physical properties of the resulting detergent particles were as listed in Table 4.

[0157] Incidentally, in Table 4, in addition to an average particle size (entire particles) of the detergent particles, an average particle size of the detergent particles that passed through the sieve having an opening of 1180 μm used in the calculation of yield was also listed together. The free-flowability, the bulk density, and the dissolution ratio of the detergent particles, and the bleed-out property of the component c) were determined and/or evaluated using detergent particles which were allowed to pass through the above-mentioned sieve to exclude aggregated or coarse particles.

Comparative Example 2-1

[0158] Detergent particles were obtained in the same manner as in Example 2-1 with the components listed in Table

4, using a base particle substitute powder in place of the base particles. Here, in Comparative Example 2-1, as the base particle substitute powder, a powder produced by dry-blending the components so as to have the ratio of the powder raw material blended in the base particles in a given compositional ratio was used. The physical properties of the resulting detergent particles are shown in Table 4. Detergent particles having excellent free-flowability were obtained in the same manner as in Examples 2-1 to 2-9; however, the amount of a modifying agent fine powder (zeolite) which was necessary to obtain a detergent having excellent free-flowability was dramatically increased as compared to those of Examples 2-1 to 2-9. Also, the aggregation and formation of coarse particles of the particles took place, thereby dramatically lowering its yield. In addition, its dissolution ratio was lowered.

[0159] The amount and yield of the fine powder (zeolite) added at this time, and the average particle size, the free-flowability, the bulk density, and the dissolution ratio of the detergent particles are shown in Table 4.

[0160] Here, the surfactant composition used in Comparative Example 2-1 was the same one as that used in Example 2-1. The components and water content and viscosity are as shown in Table 3.

Comparative Example 2-2

[0161] Detergent particles were obtained in the same manner as in Example 2-1 with the components listed in Table 4, using a base particle substitute powder in the same manner as in Comparative Example 2-1. Here, in Comparative Example 2-2, as the base particle substitute powder, a powder produced by dry-blending sodium bicarbonate and LIGHT ASH in a ratio of sodium bicarbonate / LIGHT ASH =2/1 was used. The physical properties of the resulting detergent particles are shown in Table 4. The amount of a modifying agent fine powder (zeolite) which was necessary to improve its free-flowability was dramatically increased as compared to those of Examples 2-1 to 2-9. Also, the aggregation and formation of coarse particles of the particles took place, thereby dramatically lowering its yield. The amount and yield of the fine powder added at this time, and the average particle size, the free-flowability, the bulk density, and the dissolution ratio of the detergent particles are shown in Table 4.

[0162] Here, the surfactant composition used in Comparative Example 2-2 was the same one as that used in Example 2-1.

[0163] It can be seen from the results of Table 2 that all of the detergent particles obtained in Examples 2-1 to 2-9 are excellent in free-flowability, dissolution ratio, and yield, as compared to those of Comparative Examples 2-1 and 2-2.

Example 3

[0164] Base particles used in Examples 3-1 and 3-2 were produced by the following procedures. The amount 495 kg of water was added to a 1 m³-mixing vessel having agitation impellers. After the water temperature reached 55°C, 218 kg of sodium sulfate was added thereto. After agitating the mixture for 10 minutes, 168 kg of a 40% by weight-aqueous sodium polyacrylate solution was added thereto. After agitating the mixture for additional 10 minutes, 45 kg of sodium chloride and 220 kg of zeolite were added thereto, and the resulting mixture was agitated for 30 minutes, to obtain a homogeneous slurry. The final temperature of this slurry was 58°C.

[0165] This slurry was sprayed at a spraying pressure of 25 kg/cm² from a pressure spray nozzle arranged near the top of a spray-drying tower. A high-temperature gas to be fed to the spray-drying tower was supplied at a temperature of 225°C to the bottom of the tower and exhausted at a temperature of 105°C from the top of the tower. The water content of the base particles was 2.5%.

[0166] The resulting base particles had physical properties such that the base particles had an average particle size of 192 μm, a bulk density of 536 g/L, a free-flowability of 5.2 seconds, and a supporting ability of 45 mL/100 g.

[0167] In addition, the components of the surfactant composition used in Examples 3-1 to 3-2 are those as listed in Table 5.

Example 3-1

[0168] The powder raw material composed of 100 parts by weight of the base particles previously heated to 50°C was supplied into Lödige Mixer (manufactured by MATSUBO CORPORATION; capacity: 20 L, equipped with a jacket), and the rotation of a main shaft was started (rotational speed of main shaft: 80 r/min, Froude number of agitation impellers: 1.07). Incidentally, hot water at 80°C was allowed to flow through the jacket at 10 L/minute, without rotating a chopper (equipped with disintegration impellers). After agitating the components with the rotation of the main shaft for 1 minute, 22 parts by weight of the polyoxyethylene alkyl ether at 60°C was supplied over 1 minute, and subsequently 31 parts by weight of a surfactant composition at 60°C was supplied over 1 minute, and the components were then mixed for 6 minutes. The rotations were temporarily stopped, and 20 parts by weight of a fine powder (zeolite) was supplied to the mixer. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft

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was carried out for additional 30 seconds, and the resulting detergent particles were discharged. The physical properties of the resulting detergent particles were as listed in Table 6.

[0169] Incidentally, in Table 6, in addition to an average particle size (entire particles) of the detergent particles, an average particle size of the detergent particles that passed through the sieve having an opening of 1180 μm used in the calculation of yield was also listed together. The free-flowability, the bulk density, and the dissolution ratio of the detergent particles, and the bleed-out property of the component c) were determined and/or evaluated using detergent particles which were allowed to pass through the above-mentioned sieve to exclude aggregated or coarse particles.

[0170]

[Table 5]

Surfactant Composition (Parts by Weight)		Ex.	
		3-1	3-2
	$\begin{array}{c} \text{R-CH-COOCH}_3 \\ \\ \text{a) SO}_3\text{Na} \\ (\text{R: C14/16=65:35}) \end{array}$	100	100
	b) Water	41	41
Viscosity of Surfactant Composition [Pa·s] (60°C)		5	5

[0171]

[Table 6]

Components of Detergent Particles			Ex.		
			3-1	3-2	
	<u>Surfactant Composition</u>		31	34	
	c) Polyoxyethylene Alkyl Ether		22		
	Polyethylene Glycol				
	Fatty Acid				
	Base Particles		100	100	
	Base Particle Substitute				
	Raw Material Powder				
		Sodium Carbonate			
		Crystalline Silicate			
	<u>Fine Powder</u>				
		Crystalline Silicate			
		Zeolite		20	34
		Sodium Tripolyphosphate			
Average Particle Size of Detergent Particles [μm]			225	211	
Yield [%]			98	99	
Degree of Particle Growth [-]			1.17	1.05	
Average Particle Size of Detergent Particles [μm]			223	210	
Free-Flowability of Detergent Particles [s]			6.2	6.1	
Bulk Density of Detergent Particles [g/L]			694	651	
Dissolution Ratio of Detergent Particles [%]			95	97	

(continued)

Components of Detergent Particles	Ex.	
	3-1	3-2
Bleed-out Property of Component c)	2	-

[0172] In Tables 5 and 6, the followings were used.

Zeolite: manufactured by Zeobuilder under the trade name of Zeobuilder (zeolite 4A-type, average particle size 3.5 μm); and
 Polyoxyethylene Alkyl Ether: manufactured by Kao Corporation under the trade name of 108KM (average number of moles of ethylene oxide: 8.5, number of carbon atoms of alkyl moiety: 12-14), melting point: 18°C).

Example 3-2

[0173] The powder raw material composed of 100 parts by weight of the base particles previously heated to 50°C was supplied into Lödige Mixer (manufactured by MATSUBO CORPORATION; capacity: 20 L, equipped with a jacket), and the rotation of a main shaft was started (rotational speed of main shaft: 80 r/min, Froude number of agitation impellers: 1.07). Incidentally, hot water at 80°C was allowed to flow through the jacket at 10 L/minute, without rotating a chopper (equipped with disintegration impellers). After agitating the components with the rotation of the main shaft for 1 minute, 34 parts by weight of a surfactant composition at 60°C was supplied over 1 minute, and the components were then mixed for 6 minutes. The rotations were temporarily stopped, and 34 parts by weight of a fine powder (zeolite) was supplied to the mixer. The rotations of the main shaft (rotational speed: 150 r/min, Froude number of agitation impellers: 3.8) and the chopper (rotational speed of chopper: 3600 r/min, Froude number of disintegration impellers: 1010) were carried out for 15 seconds. After the 15 seconds, the rotation of the chopper was stopped, and the rotation only with the main shaft was carried out for additional 30 seconds, and the resulting detergent particles were discharged. The physical properties of the resulting detergent particles were as listed in Table 6.

[0174] Here, the surfactant composition used in Example 3-2 was the same one as that used in Example 3-2. The components and water content and viscosity are as shown in Table 5.

INDUSTRIAL APPLICABILITY

[0175] The uni-core detergent particles of the present invention can be suitably used in, for example, the production of a laundry detergent, a dishwashing detergent, or the like.

Claims

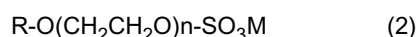
1. A method for producing uni-core detergent particles having an average particle size of 150 μm or more and a degree of particle growth of 1.5 or less comprising the steps of:

step A): preparing a surfactant composition comprising:

a) an anionic surfactant represented by any of the following formulae (1) to (3):



wherein R is an alkyl group or an alkenyl group having 10 to 18 carbon atoms; and M is an alkali metal atom or an amine,



wherein R is an alkyl group or an alkenyl group having 10 to 18 carbon atoms; n is an average number of moles added of from 0.1 to 3.0; and M is an alkali metal atom, or an ammonium or an organic amine, and



wherein R is an alkyl group or an alkenyl group having 4 to 22 carbon atoms; M is an alkali metal atom, an alkaline earth metal atom, an alkanolamine or an ammonium; and A is an alkyl group having 1 to 4 carbon atoms, H, or M, and

b) water in an amount of from 25 to 65 parts by weight based on 100 parts by weight of the said component a);

step B): mixing the surfactant composition obtained in step A) and base particles having a supporting ability of 20 mL/100 g or more and comprising a water-soluble inorganic salt produced by spray-drying, while substantially maintaining the form of the base particles; and

step C): surface-modifying the mixture obtained in step B) with a fine powder.

2. The method for producing uni-core detergent particles according to claim 1, wherein the component a) is contained in an amount of from 5 to 30% by weight of the detergent particles.
3. The method for producing uni-core detergent particles according to claim 1 or 2, wherein the detergent particles further comprise c) a nonionic surfactant having a melting point of 30°C or lower in the range of from 1 to 20% by weight of the detergent particles.
4. The method for producing uni-core detergent particles according to claim 3, wherein the component c) is mixed with the base particles prior to the surfactant composition prepared in step A).

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2005/014453

A. CLASSIFICATION OF SUBJECT MATTER

C11D11/00 (2006.01), **C11D1/29** (2006.01), **C11D3/04** (2006.01), **C11D11/02** (2006.01), **C11D17/06** (2006.01)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C11D11/00 (2006.01), **C11D1/29** (2006.01), **C11D3/04** (2006.01), **C11D11/02** (2006.01), **C11D17/06** (2006.01)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho	1922-1996	Jitsuyo Shinan Toroku Koho	1996-2005
Kokai Jitsuyo Shinan Koho	1971-2005	Toroku Jitsuyo Shinan Koho	1994-2005

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	WO 00/77158 A1 (Kao Corp.), 21 December, 2000 (21.12.00), Claims 1 to 11; page 4; pages 10 to 19; page 15, line 16 to page 17, line 1; page 19, lines 12 to 21 & JP 2004-244644 A & US 6864221 B1 & EP 1104806 A1 & EP 1104803 A1 & WO 00/77148 A1 & CN 1320157 A & CN 1320156 A & AU 744708 B & AU 5247300 A	1-4 4
X Y	JP 2004-210956 A (Kao Corp.), 29 July, 2004 (29.07.04), Claims 1 to 6; Par. Nos. [0032], [0054] to [0059], [0070]; table 1 (Family: none)	1-3 4

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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"&" document member of the same patent family

Date of the actual completion of the international search
25 October, 2005 (25.10.05)

Date of mailing of the international search report
08 November, 2005 (08.11.05)

Name and mailing address of the ISA/
Japanese Patent Office

Authorized officer

Facsimile No.

Telephone No.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2005/014453

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	JP 2001-3095 A (Kao Corp.), 09 January, 2001 (09.01.01), Claim 1; Par. Nos. [0006], [0017], [0025], [0037], [0039]; examples (Family: none)	1-3 4
X Y	JP 2000-351999 A (Kao Corp.), 19 December, 2000 (19.12.00), Claim 1; Par. Nos. [0011], [0040], [0043], [0046], [0049] to [0050], [0055]	1-3 4
X Y	JP 2000-355698 A (Kao Corp.), 26 December, 2000 (26.12.00), Claim 1; Par. Nos. [0038] to [0042], [0045], [0077] & US 6645931 B1 & US 2004-0127387 A1 & EP 1061126 A1 & DE 60001769 T1	1-3 4
X Y	WO 99/29830 A1 (Kao Corp.), 17 June, 1999 (17.06.99), Claims 1 to 11; page 16; pages 12 to 15; page 16, line 22 to page 17, line 5; page 18, lines 7 to 18; page 19, line 17 to page 20, line 1; page 22, lines 20 to 23; table 1 & JP 3123757 B1 & US 2002-155977 A1 & US 6376453 B1 & EP 969082 A1 & WO 99/29829 A1 & CN 1252095 A & AU 744709 B	1-3 4

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REFERENCES CITED IN THE DESCRIPTION

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