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Susuki et al.

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[54] **DETERGENT COMPOSED OF HOLLOW SPHERICAL PELLETS, AND PROCESS FOR MANUFACTURING THE SAME**

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 94,420, Dec. 2, 1970, abandoned.

[30] **Foreign Application Priority Data**

Dec. 3, 1969 Japan 44-97293

[52] **U.S. Cl.** **252/531;** 252/135; 252/174; 252/536; 252/539; 264/53; 264/141

[51] **Int. Cl.** ... **C11d 3/08;** C11d 11/00; C11d 13/20

[58] **Field of Search** 252/135, 531, 536, 539, 252/174; 423/332; 264/141, 142, 143, 42, 53

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[57] **ABSTRACT**

This invention concerns detergents composed of hollow spherical pellets which have various advantages such as small apparent specific gravity, moderate congeability even at a high temperature and humidity, uniformity in diameter, and extremely high mechanical strength as well as high water solubility, which are obtained by pelletizing a detergent composition being in a plastic state, and containing mainly sodium silicate, surface active agent(s), water, and inorganic builders, followed by thermally drying and foaming said composition. Further, this invention provides an effective process for manufacturing such detergents.

7 Claims, 5 Drawing Figures

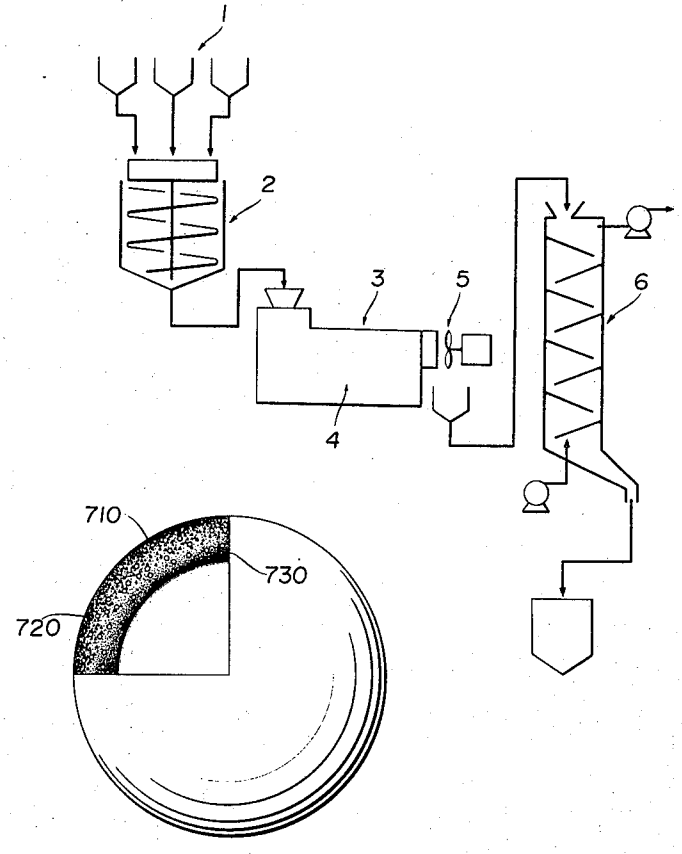


FIG. 1

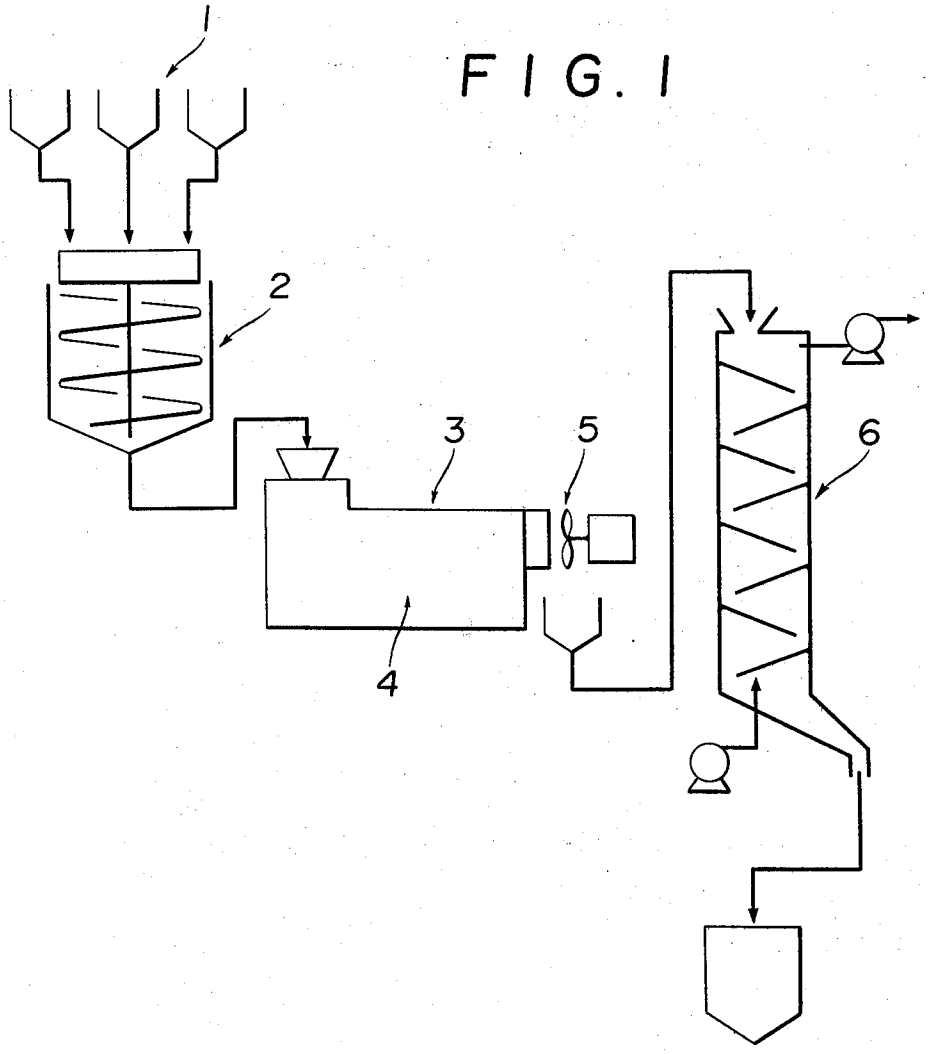


FIG. 2

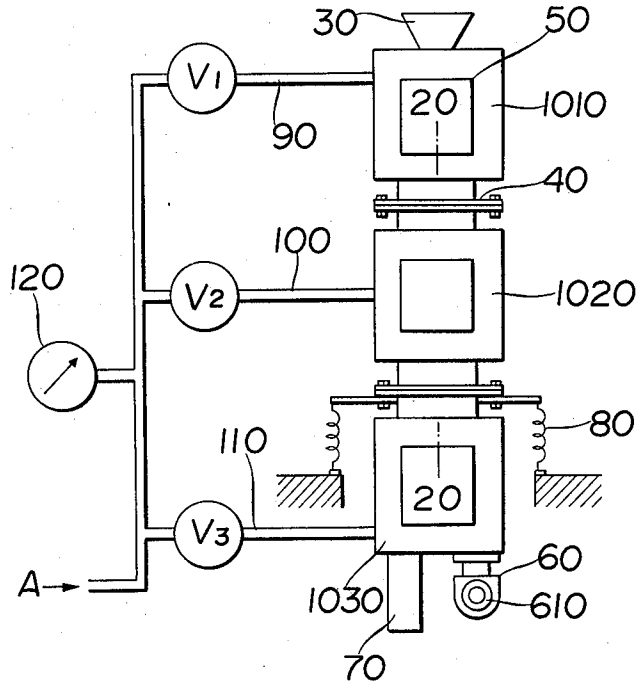


FIG. 3

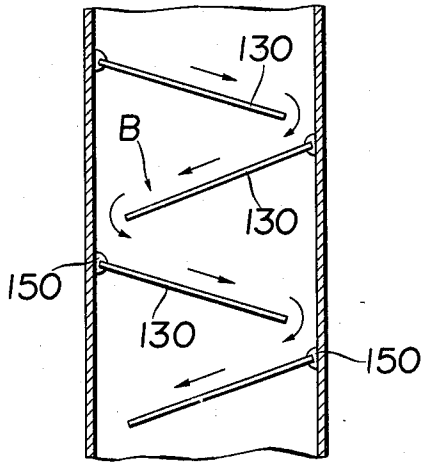


FIG. 4

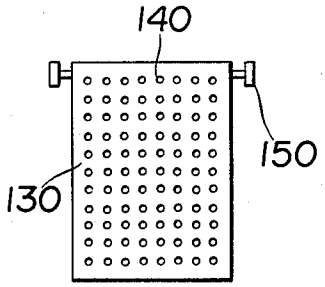
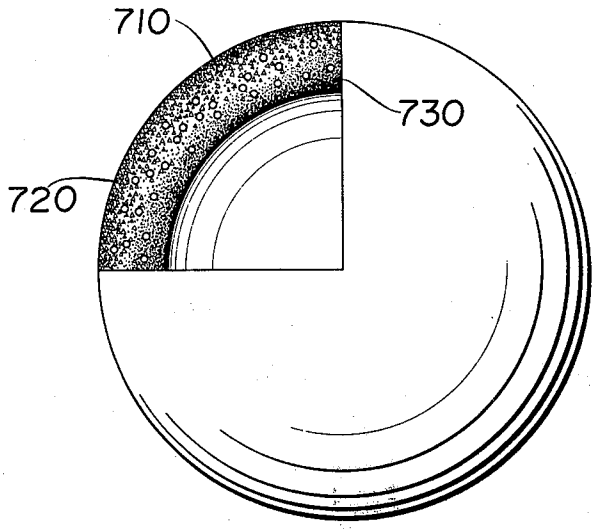


FIG. 5



DETERGENT COMPOSED OF HOLLOW SPHERICAL PELLETS, AND PROCESS FOR MANUFACTURING THE SAME

This invention is a continuation-in-part application of Ser. No. 94,420 filed on Dec. 2, 1970 now abandoned.

BACKGROUND OF THE INVENTION

a. Field of the Invention

This invention relates to detergents composed of hollow spherical pellets, and particularly to synthetic detergents consisting of hollow spherical pellets which comprise a matrix of sodium silicate, surface active agent(s), and water, and dispersoid material(s) such as Glauber's salt, sodium tripolyphosphate or other inorganic materials evenly distributed throughout said matrix in a suspended state, and to a process for manufacturing said detergents.

b. Description of the Prior Art

Conventionally, the synthetic detergents commonly used in washers and the like were shaped in granules. However, those granular detergents had various weekpoints such as small mechanical strength and unhomogeneity in diameter of the component granules, which rendered the detergents easy to get powdered. As a consequence, the conventional detergents not only tended to irritate operator's or consumers' eyes and noses but also caused subsidence or reduction in their own volumes in storage or transit.

Further, the conventional detergents, due to their granular shapes, were apt to agglomerate upon being thrown into water, and were not so easily dissolved in water. In addition, they had so high moisture permiability as to coagulate at a high temperature and humidity.

While, as to the process for manufacturing such conventional detergents, the conventionally prevailing method comprised a spray-drying process of forming synthetic detergents which consisted of steps by spray-drying in a hot air stream heated by combustion a slurry of a detergent composition containing a surface active agent such as sulfate esters of a higher alcohol or alkylbenzenesulfonates, builders such as sodium phosphate, sodium carbonate, and sodium sulfate and/or other additives, and 40 – 60 percent by weight of water.

While the above process is suitable for mass production preferably providing homogeneity of the detergents, there is a disadvantage that in order to make easier the spray-drying operation some water has to be added to the slurry to adjust the viscosity thereof, and thereafter a relatively greater part of said water must be evaporated later.

Another disadvantage is that the proportion of sodium silicate to be incorporated in the slurry is required to be limited despite the fact that sodium silicate is useful for decreasing the production cost as well as increasing the detergency; when the slurry is spray-dried, the sodium silicate contained in the slurry reacts with CO_2 in the combustion gas, thereby producing water-insoluble silicate which makes washing water muddy.

Further, the above conventional method requires relatively large-scaled and therefore expensive manufacturing equipment.

SUMMARY OF THE INVENTION

This invention aims to provide a new type of detergents which are composed of hollow spherical pellets instead of granules, and therefore are free from the above-mentioned disadvantages possessed by the conventional type of detergents.

Another object of this invention is to provide detergents composed of hollow spherical pellets which are uniform in diameter, high in mechanical strength as well as in modulus of elasticity so that they can have high measurability but show little subsidence or reduction in apparent volume during storage or transit.

A further object of this invention is to provide detergents composed of hollow spherical pellets which are low in apparent specific gravity, and high water-solubility so that they disperse immediately upon being thrown in washing water without floating over the water surface in an agglomerated state for a long time.

Still another object of this invention is to provide detergents composed of hollow spherical pellets which exhibit moderate congeability even at a high temperature and humidity, and accordingly has little viscosity and high fluidity.

The above-mentioned objects are obtained by mixing and kneading at a temperature of between 40° – 80°C a detergent composition essentially consisting of a matrix of sodium silicate, surface active agent(s), and water, and dispersoid materials such as inorganic builders of known types, into a plastic mass, and then pelletizing said mass by mechanical means, followed by thermally foaming the pellets thus obtained at a temperature of between 150° – 300°C in a drying device.

In a detergent pellet of this invention which is physicochemically in such a state that some of the individual ingredients are suspended in the other ingredients, the "matrix" serves as a dispersion medium, while the "dispersoid materials" as inorganic substances being suspended in said matrix and adapted for use as builders in the detergent composition.

The compounding ratio of the individual ingredients which constitute the matrix of the plastic detergent composition is as follows: 30 – 50 percent by weight of sodium silicate, 15 – 30 percent by weight of surface active agent(s), and 30 – 50 percent by weight of water. The plastic detergent composition is obtained by mixing 40 – 75 percent by weight of said matrix incorporated with 60 – 25 percent by weight of said dispersoid materials.

As a sodium silicate adapted for the purpose of this invention, metalsilicate is useful, and more specifically such metalsilicate is preferable as contains a metal oxide consisting of alkali metals like sodium or potasium. Particularly out of these alkali metals, sodium oxides are most preferable from the viewpoint of the quality of the final products and for economical advantage. Further, in this case, the most preferable ratio of Na_2O to SiO_2 in sodium silicate should be 1 : 2.1 – 3.1.

The surface active agents in this invention can be organic ester sulfonate of higher alcohols such as coconut oil reduction alcohol or sperm oil, or various organic sulfonate such as alkyl aryl sulfonate (those of which the alkyl group has 8 – 16 molecules of carbon), alkane sulfonate (those of which the alkane group has 8 – 24 molecules of carbon), or alkene sulfonate (those of

which the alkene group has 8 – 24 molecules of carbon).

The above-mentioned dispersoid materials may be inorganic builders such as sodium tripolyphosphate, sodium sulfate, or sodium carbonate.

These materials will serve to enable the non-foamed pellets of the detergent to have plasticity enough to maintain their shapes just prior to thermal foaming to be described hereinafter. Further, in order to decrease the apparent specific gravity of the product obtained, foaming agent(s) may be added. The foaming agents available are generally toluenesulfonylhydrazide, azocarbonyl acid amide and other nitrogen supplying foaming agents and are most effective when used in the range between 0.1 and 5 percent. Further, without losing the essential characteristics of the detergent compositions, various additives can be added to the above detergent composition such as bactericide, fungicide, anti-discolouring agent, dye, optical bleaching agent, perfume, lathering agent (ethanolamine), anti-dirt absorption agent (carboxymethylcellulose, polyvinyl alcohol, etc.), lather restrainer, skin protection agent, etc.

The above-described plastic detergent composition is useful for producing damp pellets of any size ranging between 0.6 and 3.0 mm diameter when the detergent composition is mixed in a Henschel mixer which is provided with a jacket type cooling tower or other appropriate cooling device on the outer part thereof, and is thrown into a pelletizing device. Then, a pelletized detergent of hollow spherical pellets is obtained by thermally drying said damp pellets with the appropriate thermal drying device available.

This invention will be more explicitly understood by the following detailed description while in reference to the accompanying drawings:

FIG. 1 shows illustratively an example of the apparatus for producing the detergent consisting of hollow spherical pellets.

FIG. 2 shows an example of the drying device for continuously drying the damp pellets of the detergent.

FIG. 3 shows a cross sectional view taken along the line **20** — **20** in the drying device of FIG. **2**,

FIG. 4 shows a plan view viewed from the arrow B in FIG. **3**, and

FIG. 5 shows a pellet of the detergent according to the invention, partly broken away.

DETAILED DESCRIPTION OF THE INVENTION

FIG. **1** shows diagrammatically an example of the apparatus for producing the detergent of hollow spherical pellets from the slurry. In FIG. **1**, the steps for obtaining a damp pelletized detergent from the slurry are as follows: Numeral **1** designates a measuring means for measuring the volumes of sodium silicate, surface active agent(s), water, and inorganic builders, etc. being components for the plastic detergent composition, and the delivering these components into a mixer **2.** In the mixer **2,** are kneaded at a temperature of 40° – 80°C and for 10 – 20 minutes the components of a detergent composition comprising 40 – 75 percent by weight of the matrix ingredients consisting of sodium silicate, surface active agent(s), and water, and 60 – 25 percent by weight of other ingredients consisting of inorganic builders and or additives. Thus, said kneaded components are changed into a plastic mass.

The temperature in the mixer should be adjusted within the above range since the plastic mass or detergent composition would be liable to be foamed at higher than 80°C while it would easily change into flakes at lower than 40°C. After being kneaded, the plastic mass is fed into a pelletizer **3** comprising an extruder **4** and a rotary cutter **5.** At the exit of the extruder **4** is provided a cutting plate having a plurality of bores arranged over the surface thereof like a honeycomb, through which bores are extruded out the kneaded plastic mass under a pressure ranging between 30 – 60 kgs/cm^2, and at a temperature of 40° – 80°C. Then, the plastic mass extruded out through the bores is cut by the rotary cutter **5** to a desired length.

The cutting plate may be cooled to lower than 40°C in order to effectively prevent the pellets from agglomerating into a mass.

The pellets thus obtained are moderately congealed, fully plastic, and completely free from agglomerating nor agglutinating with one another.

Next, the pellets thus formed are thrown into a drying device **6** where they are thermally foamed into hollow spherical shapes respectively as featured by the present invention.

The preferred drying device in this invention is shown in FIGS. **2, 3** and **4** and the entire device is in the form of a hollow pillar-like drying tower which is vertically arranged as shown in FIG. **2.** The drying tower comprises an indefinite number of unit drying chambers (in FIG. **2,** three chambers **1010, 1020, 1030** supported by a supporting device **80** loaded with a coiled-spring) which are interconnected by a flange **40** provided in each unit drying chamber.

This drying tower is mounted through a coil-spring-like supporting device **80.** At an appropriate place of the drying tower or the lower end thereof, is mounted a vibrator **60** designed to rotate an eccentric weight **610.**

In the unit drying chamber **1010** of the upper part of the drying tower, a blast pipe **90** is connectedly provided through a valve V_1 while in the intermediate drying chamber **1020,** a blast pipe **100** is provided through a valve V_2, and, finally in the drying chamber **1030** a blast pipe **110** is connected through a valve V_3. Drying hot air is carried to the blast pipes from the direction of the Arrow A. Numeral **120** designates a pressure gauge.

The interior of the drying tower is shown in cross-section in FIG. **3.** As shown in FIG. **4,** sieves **130** having a number of small holes **140** are mounted through hinges **150** on the interior of the drying tower, being inclined downward respectively and arranged alternately on the opposite inner walls of the drying tower in a descending manner.

This incline is designed to be moved wholly by the rod (not shown) connected to each sieve and to be adjusted freely. In the examples to be described hereafter, it is preferable to adjust the area ratio of holes **140** in the sieve **130** within the range of 10 to 50 percent and adjust the incline angle within the range of 10° to 40°. Numeral **30** shows a hopper which is the in-feed station for the pellets, numeral **70** the out-feed station and numeral **50** a window for observation provided in each unit drying chamber.

The process for continuously drying the damp pellets by means of the aforedescribed apparatus will be understood with an explanation below-mentioned. The

pellets to be dried is continuously supplied from the infeed station **30.** The pellets move downward with their own gravity, while rolling on the sieves **130.** The vibration actuated by the vibrator **60** is effective in causing a movement of the rolling. While the pellets are rolling and moving downward, each pellet becomes round, less angular and uniform in grain size.

The hot blast for drying is usually employed at a temperature of 150° and 400°C, being supplied from the direction of the arrow A. When the valve V_1 opens, while the valves V_2 and V_3 close, the hot air streams from upward to downward in the same direction as the downward movement of the objects to be dried. When the valve V_3 opens, while the valves V_1, V_2 close, the hot air streams in an opposite direction to the downward movement of these objects to be dried. When the intermediate valve V_2 opens while either valve V_1 or V_3 closes, the hot air is a combination of these two procedures. Thus, the different methods of effectuating blasts of the hot air are characterized by the fact that the same direction stream dries the pellets quickly, thereby obtaining pelletized detergents with a smaller apparent specific gravity, a thinner film surface and an excellent water-solubility. On the other hand, the opposing direction stream dries the pellets slowly, thereby obtaining pelletized detergents with a smoother surface and a larger apparent specific density. Further, the combination type of stream obtains a desired apparent specific gravity and allows free control of the drying speed of the pellets.

The process and apparatus in accordance with this invention easily allows the sieves to be adjusted in an incline angle thereof and can easily change the number of connecting unit drying chambers thereby adjusting the hours of retention of the pellets in the drying tower. Hence, the best drying may be selected according to the starting water content in the pellets.

A further advantage is that the drying state of the pellets can be conveniently observed through the observation window **50,** thereby easily meeting any unexpected situation.

Now, description will be made on the process for producing the detergents consisting of hollow spherical pellets from the plastic detergent composition according to the above-described drying method: The matrix of this invention which contains sodium silicate, surface active agent(s), and water, due to its air-impermeability, is gradually foamed in the drying device by evaporation of water contained therein, so that the pellets soften to become spherical. As they are further foamed, the individual gassed cells in each pellet get larger so that they cojoin one another to form a single large cell. Simultaneously, the pellets are dried faster towards the outer portions thereof to become hollow spherical pellets which compose the detergent of this invention as shown in FIG. **5.**

In the above-mentioned thermal foaming and drying process, a test proved that a change occurs in the distribution of sodium silicate and surface active agents in the pellet: As seen in FIG. **5,** the outer portion of the shell of a pellet has a larger proportion of sodium silicate **720** and a smaller proportion of surface active agent(s), while the inner portion thereof has a larger proportion of surface active agent(s) **730** and a smaller proportion of sodium silicate. But, the additives such as inorganic builders are distributed evenly throughout the entire shell. Meanwhile, numeral **710** represents bubbles scattered throughout the interior of the shell. It should be noted that the detergent of this invention having the above characteristics are never obtainable according to the conventional spray-drying methods, but available solely by means of the process according to this invention.

The size of the pellets according to this invention can be freely selected within the range between 1 and 5mm, and the most preferable size is 1.5 – 2.5mm which is appropriate for practical use. Particularly, those pellets obtained by extruding through a single cutting plate are extremely uniform in diameter. The thickness of the shells of the individual pellets ranges between 2 – 50 percent of the radius of the pellets, of which the most preferable percentage is 10 – 30 percent. The hollow pellets have a rather small specific gravity ranging between 0.1 – 0.4, preferably between 0.15 – 0.3.

As mentioned above, the pellets of this invention are incorporated with a larger proportion of sodium silicate towards the outer portions of the shells thereof while a larger proportion of surface active agents towards the inner portions thereof, so that they can have a high modulus of elasticity; their restitution coefficient ranges between 0.1 – 0.6. While, as to the mechanical strength of the individual pellets, a test on the breaking load value of the pellets found that they could have a mechanical strength of 5 – 200gr. per piece, which value is most desirable for the handling purpose. Further, another test was made on the powdering property of the pellets which comprised steps of bottling them, and then vertically vibrating the bottle for 30 minutes at 300 reciprocations per minute and with a stroke of 100mm, with the result that just few pellets got powdered. These tests explicitly show the considerably high mechanical strength of the detergent of hollow spherical pellets according to this invention.

The uniformity in diameter of the individual pellets brings about further advantages such as almost little subsidence or reduction in apparent volume of the pellets in reduction high measurability, and excellent flow characteristics due to their small viscosity (A test has shown that they have an angle of repose between 3° – 20°).

Such pelletized detergent according to this invention, once thrown in water, will exhibit much higher water-solubility, smaller specific gravity compared with conventional granular detergents, as well as uniform thickness of the pellet shells. Further, the characteristics of the detergent exhibits a high temporary dispersibility (i.e. the extent of water-solution of the detergent floating over the water-surface without being agitated.) completely prevents the detergent from floating in lumps over the surface of water for a long time after being thrown in water or agglomerating to be precipitated down to the washer bottom. Still further, the use of not only sodium silicate but also Glauber's salt, sodium tripolyphosphate, and the like contained in the detergent as detergent builders enables the detergent to have detergency substantially equivalent to the conventional detergents.

What is more important is that the detergent of this invention has a fine appearance, and also that it will not irritate the user's eyes or nose.

EXAMPLE 1

	% by weight
Sodium silicate (Na_2O :SiO_2 = 1 : 2.1)	28.8
Sodium Dodecylbenzene Sulfonic Acid	14.4
Water	28.8
Sodium Tripolyphosphate	12.6
Sodium Carboxymethylcellulose	1.4
Glauber's Salt	14 .0
Total:	100.0

The above ingredients were evenly mixed and kneaded at 80°C to form a plastic mass which in turn was thrown into the pelletizer to be extruded out through the bores of the cutting plate (the bore diameter = 1.8mm) under an extruding pressure of 50kg/cm², followed by cutting the extruded slurry with the rotary cutter, thus to obtain pellets each with a diameter of 2.0mm. The pellets thus obtained were thrown into the drying device to be subjected, for one minute, to counter air current flowing in said device and having a temperature of 350°C to be foamed and dried. Thus, hollow spherical pellets containing 8 percent by weight of water were obtained. The shape and property of the obtained product were as follows:

Pellet size	3 – 3.5mm
Thickness of Shell (Average)	0.7mm
Mechanical Strength of Pellet (Average)	60gr/pce.
Restitution Coefficient (Average)	0.5
Apparent Specific Gravity	0.28
Angle of Repose	4 degrees

The obtained product was non-caking, highly fluid enough to be measurable with easiness, and so uniform in diameter as to be almost free from subsiding in storage or transit. In order to test the breaking load of the pellets, a bottle stuffed with the pellets was vertically vibrated for 30 minutes at 300 reciprocations per minute and with a stroke of 100mm, to result in less than one percentage of penetration of pellets through a Tyler's 32-mesh screen. Further, also the product turned out to have excellent moisture-impermiability as well as congeability.

Next, a test was made on the water-solubility of the product to obtain the following results:

10 grams of the present detergent was thrown into a washer of Trade Mark "Coupld D" of Mitsubishi Electric Co., Ltd., while agitating 30 litre of water at a temperature of 25°C therein, to be completely dissolved in water in 30 seconds.

There was left almost nothing water-insoluble, and were produced a considerable deal of lathers. The product proved to have detergency almost as high as that of conventional types of detergents. Further, it should be noted that the products according to this invention has so high temporary dispersibility as to be quite free from agglomerating in water.

EXAMPLES 2 – 7

In these examples, the detergents were prepared according to the same process as Example 1 but under the following condition, to obtain the below-mentioned shapes and properties:

	Example	2	3	4	5	6	7
		(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)
Composition	Sodium Silicate (Na_2O:SiO_2=1:2.1)	30.0	16.0	21.6	21.6	36.0	36.0
	Sodium Dodecyl-benzene Sulfonic Acid	15.0	8.0	14.4	21.6	10.8	14.4
	Water	30.0	16.0	36.0	28.8	25.2	21.6
	Inorganic Builders	25.0	60.0	28.0	28.0	28.0	28.0
Operating Condition		Same as Example 1	Same as Example 1	Same as Example 1	Same as Example 1	Same as Example 1	Same as Example 1
Shapes and Properties	Moisture Contents (%)	9	5	11	8	7	7
	Pellet Size(mm)	3–3.5	3–3.5	3–3.5	3–3.5	3–3.5	3–3.5
	Thickness of Shell(mm)	0.7	0.9	0.6	0.7	0.8	0.8
	Apparent Specific Gravity	0.27	0.33	0.25	0.28	0.29	0.30
	Restitution Coefficient	0.5	0.4	0.3	0.5	0.6	0.6
	Mechanical Strength of Pellet (g/pce.)	60	40	30	55	120	130
	Angle of Repose (degree)	4	4	4	4	4	4
	Duration of Water-resolution (sec.)	30	20	20	30	40	40
	Temporary Dispersibility	Good	Good	Good	Good	Good	Good
	Residue water-insoluble	None	None	None	None	None	None
	Detergency	Good	Good	Good	Good	Good	Good

Note: Inorganic builders consist of the same materials of the same proportions as Example 1.

EXAMPLE 8

In this example, was followed the same process under the same operating condition as in Example 1 except that here Sodium silicate consisting of Na_2O and SiO_2 in the mol ratio of 1 to 2.5, and Olefin Sulfonic Acid having 15 – 18 carbon atoms were used in place of Sodium silicate having a 1 to 2.1 mol proportion of Na_2O to SiO_2, and Sodium Dodecylbenzene Sulfonic Acid, respectively, thus to obtain products substantially equivalent to those obtained in Example 1.

EXAMPLE 9

In this example, was followed the same process under the same operating condition as in Example 1 except that Sodium silicate consisting of Na_2O and SiO_2 in the mol ratio of 1 to 3.0 was used instead of that in Example 1, thus to obtain products substantially equivalent to those obtained in Example 8.

EXAMPLE 10

In this example, was followed the same process under the same operating condition as in Example 1 except that here Ester of Coconut Reduction Alcohol Sulfonate was used in place of Sodium Dodecylbenzen Sulfonic Acid in Example 1, thus to obtain products substantially equivalent to those obtained Example 1.

What we claim is:

1. A process for manufacturing a detergent composed of hollow spherical pellets comprising the steps of mixing and kneading

a. 40 – 75 percent by weight of a matrix consisting of 30 – 50 percent by weight of sodium silicate (Na_2O : SiO_2 = 1 : 2.1 – 3.1), 15 – 30 percent by weight of a surface active agent selected from the group of organic sulfate esters and organic sulfonates, and 30 – 50 percent by weight of water, with

b. 25 – 60 percent by weight of a dispersoid material consisting substantially of inorganic builders into the form of a detergent composition having plasticity;

extruding said kneaded plastic composition through bores at a temperature of 40° to 80°C. and cutting into damp pellets having a diameter from 0.6 to 3.0 mm, and thermally drying and foaming said pellets to form said hollow spherical pellets, said drying and foaming being carried out by subjecting the pellets to a hot blast having a temperature up to 400°C. to heat said pellets to between 150° and 300°C. while rolling said pellets downward for a period of time sufficient to produce hollow spherical pellets 1 to 5 mm in diameter with a shell thickness 2 to 50 percent of the radius thereof and having an apparent specific gravity between 0.1 and 0.4.

2. The process according to claim **1,** whereby said plastic detergent composition further includes 0.1 – 5 percent by weight of a nitrogenous foaming agent consisting of either or both of toluensulfonyl hydrazide and azocarbonyl acid amide.

3. The process according to claim **1,** whereby in said step of thermally drying and foaming, hot air is used to dry said detergent composition in the form of pellets and streams in a direction opposite to the moving direction of said detergent composition.

4. A detergent prepared by the process of claim **1.**

5. The detergent as claimed in claim **4,** wherein said pellets have a mechanical strength of 5 – 200gr. per each pellet, and a restitution coefficient of 0.1 – 0.6.

6. The detergent as claimed in claim **4,** comprising sodium silicate and a surface active agent, whereby a larger proportion of said sodium silicate and a smaller proportion of said surface active agent are distributed towards the outer portion of the shell of each pellet, while a smaller proportion of said sodium silicate and a larger proportion of said surface active agent are distributed towards the inner portions of the shell of each pellet.

7. The detergent as claimed in claim **4,** whereby said pellets have an angle of repose ranging between 3 – 20°.

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