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- (21) Application No. 39751/77 (22) Filed 23 Sept. 1977 (19)
 (31) Convention Application No. 727 838 (32) Filed 29 Sept. 1976 in
 (33) United States of America (US)
 (44) Complete Specification published 11 Feb. 1981
 (51) INT. CL.³ C11D 1/00
 (52) Index at acceptance

C5D 6A5D2 6A5E 6B12N1 6C10 6C6 6D
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(54) BOTTLED DETERGENT COMPOSITIONS

(71) We, COLGATE-PALMOLIVE COMPANY, a Corporation organised under the laws of the State of Delaware, United States of America, of 300 Park Avenue, New York, New York 10022, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to bottled particulate detergent compositions.

Particulate detergent compositions based on synthetic organic detergents and builder salts to improve the detergency thereof are well known and have been marketed for many years. In a preferred form, such particles are rounded and may be globular or may be aggregates of small numbers of globular particles, such as those which result from spray drying of a crutcher mix of the various components thereof and subsequent screening to size. Post-spraying of liquid materials, especially heat-sensitive materials, onto the surfaces of spray dried particles is also known. However, it is often difficult to obtain free-flowing product containing a substantial proportion of a liquid or tacky synthetic organic detergent, such as a liquid nonionic detergent, and water-soluble inorganic builder salt(s). Consequently synthetic organic detergents of lesser tackiness, such as synthetic organic anionic detergent salts, for instance sodium linear tridecyl benzene sulphonate, which also contribute excellent detergency to built detergent compositions, have been used extensively. For convenience in dispensing, among other reasons, liquid detergents have been marketed, often in plastic bottles (i.e. bottles made of plastics materials), such as those with an integrally blow-moulded hollow handle. However, before the present invention it was not known to package a free-flowing, high bulk density heavy duty detergent composition in a bottle from which it could be dispensed as readily as a liquid, and without various disadvantages associated with liquid detergent compositions. Thus, the bottled particu-

late detergent compositions of this invention are not subject to separation into different phases, requiring the presence of detergently non-functional ingredients, such as hydrotropes, to prevent this undesirable action. Furthermore, because the particulate product is substantially dry, aqueous or liquid phase decomposition reactions between the components thereof do not occur and incorporation of stabilizers or the modifications of formulations to avoid materials which tend to react in an aqueous medium are obviated. Additionally, due to the pouring out of the particulate built detergent as a composition of solid particles the gels or cement-like deposits sometimes observed on the necks of dispensing bottles for liquid detergent compositions do not occur. In short, the present invention affords various advantages of particulate or powdered detergents with packaging and dispensing conveniences equal to or better than those associated with bottled liquid detergents.

In accordance with one aspect of the present invention a bottled particulate detergent composition comprises a bottle having a neck for dispensing detergent composition from the bottle, and a free-flowing particulate heavy duty detergent composition in the bottle, pourable through the neck, the particles of which comprise synthetic organic detergent and builder for the detergent, are of particle sizes such that at least 90% thereof passes through an 8 mesh screen and is retained on a 200 mesh screen (these and all mesh sizes given herein are U.S. Sieve Series), are of a bulk density of at least 0.5 g/cc and are of a flowability at least 70% of that of clean dry sand.

In various preferred embodiments the cross-sectional area of the passage through the bottle neck is in a particular range and within a certain range of proportions with respect to the bottle cross-sectional area; the bottle has a hollow handle integral with it, communicating with the main bottle volume and of certain dimensions; the bottle neck is circular in cross-section and is capped by a screw cap of particular size so as

to serve as a convenient measuring cap for the detergent composition being dispensed; the detergent particles are dust-free; and the detergent composition is of a certain type, with the particular properties mentioned being in certain more limited ranges.

Another aspect of the invention provides a simple method for filling the bottles and integral hollow handles thereof.

According to this other aspect of the invention the method comprises maintaining in an upright position a bottle of elongate horizontal cross-section having long and short major and minor horizontal axes, the long axis defining a vertical plane and the short axis defining an intersecting vertical plane, with a neck at the top of the bottle near one end of the vertical plane defined by the long horizontal axis and an integral hollow handle at the other end of this vertical plane, the hollow of which handle communicates with the main volume of the bottle, and directing particulate detergent composition downwardly through the bottle neck to the main volume of the bottle to fill the main volume of the bottle and the handle thereof, the filling of the handle being effected by flow of the particulate detergent composition downwardly into the handle when the detergent composition level rises above the handle, without tilting the bottle or directing particulate detergent into the handle, the bottle neck passageway being of a cross-sectional area in the range from 3 to 20 cm² and less than 40% of the average cross-sectional area of the bottle, the hollow handle including a portion of internal cross-sectional area in the range from 2 to 5 cm², and the particulate detergent composition being of particle sizes such that at least 90% thereof passes through a 20 mesh screen and is retained on a 200 mesh screen, the composition being of a bulk density of at least 0.5 g/cc and of a flowability at least 70% of that of clean dry sand.

The invention may be performed in various ways and some embodiments will now be described by way of example with reference to the accompanying drawings, in which:—

Figure 1 is a front elevation of a bottled (but uncapped) particulate detergent composition embodying the invention;

Figure 2 is a top plan view;

Figure 3 is a side elevation from the handle side;

Figure 4 is a sectional plan view taken along plane 4—4 of Figure 1;

Figure 5 is a fragmentary front elevation with a cap on the bottle;

Figure 6 is a photomicrograph of part of a spray dried builder bead or particle prior to post-spraying of synthetic organic detergent onto it (to make a free flowing built detergent composition), magnified as indicated by the scale thereon;

Figure 7 shows a cutaway portion of the bead of Figure 6, at a larger magnification as indicated by the scale thereon; and

Figure 8 is a partially schematic elevation of filling means for filling the bottle with free-flowing particulate detergent composition, showing a sequence of stages of the filling operation.

In Figure 1 a bottle 11, of transparent polyvinyl chloride plastic, comprises a body portion 13 having a handle 15 integrally blow-moulded with it, and a neck portion 17. While the bottle may be of various shapes, the preferred shape shown is substantially oval in general cross-section, at least in the upper and lower portions thereof, and has the neck at the top of the bottle and nearer to one end of the major horizontal axis of such oval, the handle being at or near the opposite end of that axis. The term "horizontal" refers to the case where the bottle is standing upright, as shown. Also, it is preferred that upper portion 14 of body wall 12 on the side remote from the handle 15 should be nearly vertical, i.e. from 70° to 90° from the horizontal, while the upper portion 16 on the side having the handle should have more slope, i.e. for 30° to 60° from the horizontal, for reasons which will be given later. Inside the bottle 11 are detergent composition particles 19, which are readily dispensable through the passageway 21 in the bottle neck by merely tipping the bottle and pouring the particulate material out therefrom. The parts so far described are also shown in Figures 2 and 3, where appropriate.

In Figure 4 a portion of the main volume 23 of the bottle is illustrated, as is a part of the interior passageway 25 through the handle 15. Both are filled with particulate detergent composition 19.

In Figures 1—4 the bottle is shown uncapped, but for normal sealing and shipping an internally threaded screw cap 27, shown in Figure 5, will be screwed in place on the threaded exterior of the neck 17. Normally the material of the cap on the bottle will be sufficiently resilient so that sealing washers need not be employed (a sealing bead may often suffice) but such washers may be used if desired.

In Figures 6 and 7 the light areas are reflections from the skeletal portions of the bead, both the internal and external parts of which help to strengthen it, and the dark areas show voids in the bead and at and just below the external surfaces thereof, into which voids liquid or dissolved synthetic organic detergent may be drawn after such detergent is sprayed onto or otherwise brought into contact with the beads, producing a bead surface essentially free of detergent, e.g. having detergent covering less than 10% of the external bead surface. The particular

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bead illustrated in Figures 6 and 7 is one obtained by the spray drying of a crutcher mix containing about 18.8 parts of pentasodium tripolyphosphate hexahydrate, 7.6 parts of sodium silicate ($\text{Na}_2\text{O}:\text{SiO}_2=2.4$), 28.3 parts of pentasodium tripolyphosphate (anhydrous) and 49.6 parts of water, spray dried to a moisture content of about 10%. All parts and percentages given herein are by weight.

In Figure 8 there are shown six stages in the filling of the bottle 11 with particulate built detergent composition 19. In the first of these, at the left of the Figure, a filling head 29 having an extensible filling nozzle 31 equipped with control valve 33 is positioned above an empty bottle 11, this bottle being as illustrated in Figures 1-4. In the adjacent portion of the Figure, illustrating the next stage of the filling operation, the extensible nozzle 31 has been lowered so that the bottom thereof is inside the neck 17 of the bottle 11 but flow of particulate heavy duty detergent to the bottle has not yet commenced. The next portion of the Figure illustrates the filling of particulate detergent composition 19 into the bottle 11, the level of the detergent particles being above the mid-point of the height of the handle and opening in the main volume of the bottle, as shown by level 35, while being at a lower level 37 in the passageway 25 of the handle portion 15 of the bottle. In the fourth stage the particulate detergent height has been increased further, so that it is above the top of the passageway 25 and particulate detergent 19 fills the passageway and forms a level surface 39 above it at about the same height as the particles in the main volume of the bottle. In the fifth stage the particles 19 have been filled to the final filling level 41, and in the sixth stage nozzle 31 has been withdrawn. At a subsequent stage in the filling and sealing operation, not shown, the bottle is automatically or manually capped, preferably the former.

The bottle may be of any suitable material although synthetic organic polymeric plastics materials, such as polyvinyl chloride, polymethyl methacrylate, polyethylene terephthalate, polyethylene, polypropylene, polystyrene, polyesters and polyethers, preferably reinforced with glass fibre, and nylons are preferred. Glass bottles may also be used. Preferably, the bottles are transparent, but opaque and translucent bottles may be used. An important advantage of the bottle materials mentioned is that they are substantially or completely moisture proof and usually are barriers to the transmission of organic volatile substances too, such as perfumes, even with comparatively thin walls, e.g. from about 1 to about 3 mm thick.

The bottles may be of various shapes but, as distinguished from jars and other con-

tainers, they have a neck portion which is relatively narrow and has a small opening or passage therein, compared to the average cross-sectional area of the bottle volume. Generally the cross-sectional area of the neck passageway will be less than 40% of the average bottle cross-section, preferably less than 30% thereof and more preferably, less than 25% thereof. To facilitate ready pouring and good control of dispensing the neck passageway is of a cross-sectional area in the range from 2 to 40 cm^2 preferably 3 to 20 cm^2 and more preferably 5 to 15 cm^2 . The neck will usually be oriented so that its passageway and walls are parallel to the vertical axis of the bottle, but they may be inclined, usually no more than 30° from the vertical but greater inclinations are also operative. The neck size will depend on the bottle volume, to an extent, and on the cap size desired for dispensing the detergent composition, about which more will be said later. For 1 quart or 1 litre size bottles, neck heights of 1 to 5 cm can be used, but normally the neck height will be from 1.5 to 3 cm. For larger sizes, such as $\frac{1}{2}$ gallon, 2 litres, gallon and 4 litre bottles, the necks will generally be 1 to 5 cm high too, but preferably from 2 to 5 cm. Non-metric units of volume referred to herein are U.S. units, e.g. "gallon" means a U.S. gallon.

Various cross-sectional shapes may be employed for the bottle but the most preferred cross-section is one which is substantially oval, as illustrated. In some cases it may be desirable to square off the rounded minor curves, and such squared off shapes, and elliptical shapes, are deemed to be oval within the meaning of that word as it is employed in this specification. Instead of oval cross-sections, other shapes may be employed, such as circular, regular polygonal, e.g. rectangular or square, and polygonal with rounded corners.

Although not essential, an integral hollow handle, which may be blow moulded or otherwise suitably moulded integrally with the neck and main body portions of the bottle, is desirable. The interior of the handle communicates with other interior portions of the bottle so that the particulate detergent composition may be filled into the handle and may flow from it. The handle will normally be located at a side of the bottle remote from the neck, and preferably will not project beyond the normal bottle wall. In other words, the handle and its finger opening lie within the bottle's general outline. The hollow handle does not have to be of regular cross-section or interior passageway area but a substantially regular or uniform passageway cross-section is preferred. The hollow handle will generally have at least a portion thereof with an internal cross-sectional area in the range of 1 to 10 cm^2 , preferably 2 to 5 cm^2 .

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Normally the handle will be of a convenient vertical length or height so that it can be grasped easily, which length has been determined to be from about 8 to 12 cm, preferably

5 9 to 11 cm. Such a height of hollow handle will be readily filled by the particulate detergent composition, and the detergent composition will flow from it easily as it is dispensed. In this respect, it is desirable to have
10 a free height, above the top of the handle passageway, between the desired fill level of the detergent composition particles and the handle passageway top, of at least 1 cm and preferably at least 5 cm, heights up to 10 or
15 15 cm being useful with large containers. The slope of the wall 16 is preferably greater than or about the same as the normal angle of repose of the particulate detergent composition after depositing during filling or
20 after dispensing, and accordingly facilitates filling of the handle passageway and discharge therefrom.

The free-flowing particulate detergent composition inside the bottle preferably has a
25 high bulk density and such particle size distribution as to promote flow and venting of gas, thereby minimizing the tendency to bridge in the container or in restricted portions thereof. The detergent composition comprises a synthetic organic detergent
30 and a builder for the detergent, and may include various adjuvant materials normally present and desirable in detergent compositions and not of such properties as to make the composition unsuitable for the present
35 purposes.

The synthetic organic detergent may be of the anionic, nonionic, cationic, ampholytic or amphoteric types, but of these the anionics
40 and nonionics materials are preferred, the nonionic generally being most suited for the present compositions although mixtures of nonionic and anionic detergents are sometimes even more preferred. Usually, no
45 cationic detergent will be present, especially when anionics are present.

The nonionic detergents can be liquid or semi-solid at room temperature and usually will be liquid or tacky at some temperature
50 below 40°C. Preferred nonionic detergents comprise ethoxylated aliphatic alcohols having straight or branched chains (preferably straight chained) of from 8 to 22 carbon atoms with from 5 to 30 ethylene oxide units per
55 molecule. Particularly suitable nonionic organic detergents of such type are manufactured by Shell Chemical Company, U.S.A. and are marketed under the trade mark NEODOL. Of the various "Neodols" available, "Neodol 25-7" (12-15 carbon atom
60 chain fatty alcohol condensed with an average of 7 ethylene oxide units) and "Neodol 45-11" (14-15 carbon atom chain fatty alcohol condensed with an average of 11
65 ethylene oxide units) are particularly pre-

ferred. Another suitable class of ethoxylated aliphatic alcohol detergents is made by Continental Oil Company, U.S.A. and marketed under the trade mark ALFONIC. Of the "Alfonics" the most preferred is
70 "Alfonic 1618-65", which is a mixture of 16 to 18 carbon atom primary alcohols ethoxylated so as to contain 65 mol percent of ethylene oxide.

Additional examples of nonionic synthetic
75 organic detergents include those marketed by BASF Wyandotte, U.S.A., under the trade mark PLURONIC. Such compounds are made by the condensation of ethylene oxide with a hydrophobic base formed by condens-
80 ing propylene oxide with propylene glycol. The hydrophobic portion of the molecule has a molecular weight of from about 1,500 to about 1,800 and the addition of polyoxyethylene (or ethylene oxide) to such
85 portion increases the water solubility of the molecule as a whole, the detergent remaining liquid at room temperature up to the point where the polyoxyethylene content is about
90 50% of the total weight of the condensation product, at which it becomes solid. Also useful nonionic detergents are the polyethylene oxide condensates of alkyl phenols, such as the condensation products of such
95 compounds wherein the alkyl group contains from 6 to 12 carbon atoms, in either a straight chain or branched chain configuration, with 5 to 25 mols of ethylene oxide per mol of alkyl phenol. The alkyl substituents in
100 such compounds may be derived from polymerized propylene or may be diisobutylene, octene or nonene, for example.

Typical anionic detergents include fatty acid soaps derived from natural or synthetic
105 fatty acids of 8 to 20 carbon atoms or from their triglycerides, e.g. coconut oil, tallow, hydrogenated coconut oil, hydrogenated tallow and mixtures thereof; linear alkyl benzene sulphonates wherein the alkyl group is of 10 to 18 carbon atoms, preferably
110 12 to 15 carbon atoms, e.g. sodium linear tridecyl benzene sulphonate; paraffin sulphonates; olefin sulphonates; and other organic sulphonates and organic sulphates, in which a lipophilic group is present which normally includes a chain of 10 to 18 carbon atoms. The various nonionic compounds described may be converted to anionic compounds by
115 sulphation or sulphonation, usually the former, at terminal hydroxyls, but in such cases normally the proportion of ethylene oxide employed to make the initial nonionic condensate product will be diminished, so that from 3 to 12 mols, preferably 5 to 10
120 mols, of ethylene oxide will be present per mol of anionic detergent.

The various anionic detergents mentioned above are preferably employed as their sodium salts, although potassium salts and in some instances small proportions of

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ammonium or triethanolamine salts may also be utilized.

Representative cationic detergents usually also possess fabric softening and antibacterial properties, these properties being especially characteristic of quaternary compounds. Examples of such materials are distearyl dimethyl ammonium chloride and 2-heptadecyl-1-methyl-1-[(2-stearoylamido)ethyl]-imidazolinium methyl sulphate.

Various amphoteric detergents are also suitable. These are generally higher fatty carboxylates, phosphates, sulphates or sulphonates which contain a cationic substituent such as an amino group which is quaternized, for example with lower alkyl groups, or may have the chain thereof extended at the amino group by condensation with an alkylene oxide, e.g. ethylene oxide. Representative commercial water-soluble amphoteric organic detergents include "Deriphat 151", which is sodium N-coco-beta-aminopropionate (manufactured by General Mills, Inc., U.S.A.) and "Miranol C2M" (anhydrous acid), made by Miranol Chemical Company, Inc., U.S.A. DERIPHAT and MIRANOL are trade marks.

Further descriptions of various suitable detergents, including descriptions of classes of detergents to which those mentioned above belong, are found in McCutcheon's *Detergents and Emulsifiers*, 1973 Annual and in *Surface Active Agents*, Vol. II, by Schwartz, Perry and Berch (Interscience Publishers, 1958).

The builder for the detergent of the present compositions is preferably an inorganic material, and of these the water-soluble salts are highly preferred, particularly the phosphates. However, organic builders such as sodium citrate, sodium gluconate, trisodium nitrilotriacetate and other organic compounds known to have builder activity and which are capable of being made into a free-flowing built detergent composition may also be employed, often in mixture with inorganic materials. Usually the organic compounds will be present as the sodium or other alkali metal salts but sometimes the free acid forms may be used. Although the phosphates are highly preferred for the making of exceptionally free flowing, high bulk density particulate materials containing large quantities of synthetic organic detergent, preferably post-added to the phosphate base beads, other inorganic materials may also be employed as builders (or in combination with them) such as silicates, borates, carbonates and bicarbonates. Ion-exchanging clays or other aluminosilicate materials, which act as builders in detergent compositions, removing hardness ions from the wash water, such as type A (preferably 4A) molecular sieves and other suitable molecular sieves, may be used as builders, preferably

with a suitable phosphate, but for non-phosphate detergents may be employed with other non-phosphate builder salts, alone or with other ion-exchanging zeolites.

With respect to the phosphates utilized and the other inorganic water-soluble builder salts, normally the sodium salts will be employed but potassium salts are also useful. Specific examples of phosphate builder salts include pentasodium tripolyphosphate, other sodium tripolyphosphates including trisodium tripolyphosphate, trisodium phosphate, disodium phosphate, monosodium phosphate, tetrasodium pyrophosphate and disodium pyrophosphate. The corresponding potassium salts may also be employed but are preferably used in mixture with the sodium salts.

Preferred supplemental builder materials, which also have anti-corrosion properties in the detergent composition and help to form desirable beads when builder beads are manufactured for subsequent overspraying or post-spraying onto them of synthetic organic detergent liquid, are alkali metal silicates, usually supplied in the form of aqueous solutions containing from 40 to 60% by weight, typically about 50% by weight, of silicate solids. Such silicates are preferably sodium silicates and the $\text{Na}_2\text{O}:\text{SiO}_2$ ratio thereof will normally be from 1:1.6 to about 1:3.4, preferably 1:2 to 1:3 and most preferably about 1:2.35 or 1:2.4.

In addition to the synthetic organic detergent and the builder components, various adjuvants may be present, usually preferably incorporated in the structures of the particles either by spray drying them with the particles from a common crutcher mix, or, when they are heat-sensitive, by post-adding them. Among such adjuvants are conventional functional and aesthetic materials such as bleaches, e.g. sodium perborate; colouring agents, e.g. pigments, dyes and optical brighteners; perfumes; foam stabilizers, e.g. alkanolamides, such as lauric myristic diethanolamide; enzymes, e.g. proteases and amylases; skin protecting and conditioning agents, e.g. water-soluble proteins of low molecular weight obtained by hydrolysis of proteinaceous materials such as animal hair, hides, gelatin and collagen; foam destroyers, e.g. silicofluorides; fabric softeners, e.g. ethoxylated lanolins; bactericides, e.g. hexachlorophene; buffering agents, e.g. alkali metal acetates and bisulphates; and flow improving agents, e.g. ground clays. Additionally, filler salts such as sodium sulphate, usually anhydrous, and sodium chloride may be present but are usually best avoided.

The built detergent composition particles of the present invention will normally contain from 50 to 98% of builder and the balance synthetic organic detergent, neglecting the presence of any other materials,

including water. Considering the presences of other materials in the composition it will usually contain from 30 to 80% of builder, from 2 to 40% of synthetic organic detergent, from 0 to 20% of adjuvants, excluding fillers, from 0 to 50% of fillers (preferably omitted) and from 3 to 15% of moisture. The particulate detergent composition may be made by any suitable means, including spray drying the entire mixture of heat-stable substances, but to obtain best flowability, least dust, highest bulk density and least undesirable chemical reactions and decompositions of components it is preferred to manufacture a base bead comprising substantially only builder with (preferably) or without water and to post-add detergent to it, such as a liquid nonionic detergent or a mixture of anionic and nonionic detergents (although anionic detergent may sometimes be spray dried with the builder). Usually when the detergent composition particles are made by such preferred methods from 60 to 98% of the base beads are of detergent builder having porous outer surfaces and skeletal internal structures and from 2 to 40% is of a synthetic organic detergent material, such as one which is liquid or tacky at a temperature below 40°C and which is disposed internally within the beads so that the outer surfaces of the beads are substantially free of the detergent and therefore are free-flowing. In preferred embodiments of the invention the particulate detergent composition comprises from 70 to 95% of base beads and from 5 to 30% of synthetic organic detergent and the base beads comprise from 45 to 85% of a phosphate builder salt, from 5 to 15% of alkali metal silicate and from 5 to 15% of water. In highly preferred embodiments of the invention the synthetic organic detergent is a nonionic polyethoxylated detergent, such as one derived from aliphatic alcohol having from 8 to 22 carbon atoms in a chain therein, condensed with from 5 to 30 mols of ethylene oxide per mol of the alcohol. The phosphate salt is preferably a mixture of hydrated and anhydrous salts, the weight ratio of hydrated phosphate, usually pentasodium tripolyphosphate hexahydrate, to anhydrous phosphate, usually pentasodium tripolyphosphate, anhydrous, being in the range from 0.3:1 to 0.7:1, preferably from 0.4:1 to 0.6:1.

No matter how the particulate detergent composition is made it has been found that to produce the bottled product of this invention it is important that the final detergent composition be of particle sizes such that at least 90% thereof passes through an 8 mesh screen and is retained on a 200 mesh screen and that the particles are of a bulk density of at least 0.5 g/cc and are of a flowability of at least 70% of that of clean dry sand. Preferably at least 90% of the

detergent composition passes through a 20 mesh screen and is retained on a 200 mesh screen and more preferably over 95% of it is in the range from 40 mesh to 200 mesh, with less than 0.5% passing through a 200 mesh sieve. The bulk density is preferably in the range from 0.55 to 0.8 g/cc, and the flowability is preferably at least 75% of that of clean dry sand and may approach or equal 100%.

The particles will usually be dust free so that even after shaking in a transparent container, in a time as short as 1 or 2 seconds after cessation of shaking, the space above the detergent composition will be clear and no detergent composition will adhere to the inner walls of the container. In part, such desirable non-dusting properties are attributable to the content of nonionic detergent, which is usually present in sufficient proportion to lay dust and to prevent its being created by movement of the particles. Thus, often the amount of nonionic detergent used may be from 12 to 40% of the composition and sometimes the proportion employed will be from 20 to 40% of the composition, which can be sorbed into the interiors of the detergent particles without making the surfaces sticky so that the composition is poorly flowing. Representative of especially preferred nonionic detergents employed is the condensation product of an aliphatic alcohol having a carbon chain of 10 to 18 carbon atoms, with 6 to 14 mols of ethylene oxide per mol of the alcohol and in such case, especially when 20 to 40% is present in the detergent composition, the percentage of particulate detergent passing a 200 or 325 mesh sieve is often nil. The presence of more sorbable detergent in the particulate composition and the consequent high bulk density makes it possible to use a smaller volume of detergent composition per wash load, thereby making packaging in bottles more feasible and increasing the practicability of dispensing smaller volumes of detergent composition and measuring them in the bottle cap.

In those circumstances where dusting is noted, even very small amounts thereof, it may be desirable to coat the interior of the bottle with a thin layer of a silicone or a quaternary ammonium salt, such as those previously described, or both, to smooth the interior bottle surface and to promote diffusion of any electrostatic charge on the bottle interior and thereby obviate adhesion of the fine particles to the bottle interior. Normally the amounts of such materials employed will be sufficient to form a thin layer on the bottle interior, often only 1 to 10 molecules thick.

The cap for the bottle may be of any suitable structure and material but screw caps are preferred because of their ready avail-

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ability, trouble-free sealing and measuring capabilities. However, because the detergent composition is a particulate solid rather than a liquid completely tight sealing is not always necessary and other types of caps may be employed which do not have the sealing capacities of the normal screw caps. For example, caps that are press-fitted into place may be used, as may be those which include a slide valve to open a dispensing passage. In such and other cases the neck portion of the bottle may be of lower height than for a screw cap and may even be reduced to merely an opening in a wall of the bottle. The cap may be made of any suitable material, including synthetic organic polymeric plastics; rubber, especially hard rubbers; metals and metal alloys. Among the useful synthetic organic polymers may be mentioned melamine formaldehydes, phenol formaldehydes, nylons, polystyrenes, (dense or from foamed beads) glass fibre reinforced poysters, polypropylene and polyethylene.

The preferred internally screw threaded caps form a tight seal with the neck of the bottle when screwed into place, preventing loss of contents and preventing access of moisture of other external contaminants to the contents or transmission of moisture vapour from the bottle. Thus, because of the screw type structure and the barrier qualities of the bottle, an effective and inexpensive seal is obtained without the need for employment of special barrier liners or coatings on penetrable containers normally used for the packing of detergent powders. Additionally, although the cap is relatively small it may be used for measuring out desired quantities of the detergent composition particles. However, screw caps of a size such as to be useful for sealing off the openings in bottles of sizes such as one quart to one gallon, would not be large enough to hold in one capful, or even in several capfuls, the volume of ordinary composition detergent particles that would have to be charged to an automatic washing machine for one wash. Thus, with conventional spray dried detergent powders of a bulk density of about 0.3 g/cc onto which a small proportion of nonionic detergent component has been post-sprayed, about 90 grams or 300 cc of detergent composition particles would have to be charged to a 17 gallon washing machine tub whereas the normal bottle cap has a volume of only 5 to 15 cc. Thus, even with a volume of 15 cc one would have to measure out twenty capful of such a low density conventional powder detergent composition. This would not be acceptable to the housewife, nor would there be any advantage in following such a procedure rather than utilizing an ordinary measuring cup. However, by virtue of the present invention, utilizing a high bulk density detergent composition in a bottle with only

a slightly larger cap than is usual for such a bottle, such measurement by means of the cap is feasible. With the larger quantity of detergent, such as a liquid nonionic detergent, that may be incorporated in the builder beads it is possible to reduce by half the volume of detergent composition needed per wash load, and with beads of greater bulk density, e.g. 0.6, another halving of the volume needed is obtained. Thus, by merely making the cap slightly larger, so as to give it a volume of about 20 cc instead of 15 cc measuring out of four capful of the detergent composition powder will provide enough for one ordinary wash in a top loading machine. For side loading machines, where conventionally one uses about half as much detergent, measuring out only two capfuls will be sufficient. By further adjusting the bulk density of the composition, the synthetic organic detergent content thereof and the cap size, one may make it possible to use as little as one cap of detergent composition per wash or as much as three capful. Thus, the present invention provides a convenient means for readily measuring a quantity of particulate detergent composition, in a cap of the bottle, to provide the desired quantity of detergent composition for an ordinary wash.

Methods for the manufacture of free-flowing, high bulk density particulate detergent compositions of the desired particle sizes are known in the art and may be used in performing the invention. Thus, controlled spray drying, spray cooling, agglomeration, solidification, abrading crystalline materials, are useful. The nonionic detergent may be incorporated into base particles or may be integrally formed with the particles so long as the particles are of desired sufficiently round or rounded structure to be free-flowing and have any liquid or tacky detergent material internally held predominantly within the interstices in the particle rather than on the surface thereof (normally less than 20% will be on the surface, preferably less than 10%). After manufacture of the detergent composition particles they are filled into the bottles and the bottles are capped by the method illustrated in Figure 8 or some equivalent method, but other more complicated and less satisfactory filling techniques may also be utilized. After sealing in the bottle the shelf life of the composition is almost limitless for practical purposes, in most cases being at least three years without any undesirable changes.

The bottled composition of the invention has several advantages, some of which have already been mentioned. A comparatively inexpensive and readily obtainable bottle with a built-in hollow handle may be employed with conventional sealing means that results in the product having excellent

storage properties. The particles are rounded and of uniform shape and flow like a liquid. They are dense enough to be measurable in practical amounts in a small cap and for a multiplicity of washing quantities to be containable in a reasonably sized bottle, namely about 10 to 20 washes, normally about 12, per quart. Commercial liquid detergent compositions yield only about 8 washes per quart (such figures being for top loading washing machines). Additionally the cap can be graduated for finer measurements. The product is easily manufactured and allows the convenient incorporation of normally heat-unstable components in the detergent composition. Also, due to the fact that the composition is not in a liquid phase, stability on storage is promoted. This allows the use in the composition of certain normally less stable and more effective deterative ingredients.

The following Examples illustrate the invention.

EXAMPLE 1

An aqueous slurry is prepared consisting of 14.5 parts of pentasodium tripolyphosphate powder (anhydrous), 15.2 parts of 50% aqueous solution of sodium silicate ($\text{Na}_2\text{O}:\text{SiO}_2=1:2.4$) and 21 parts of deionized water. The slurry is brought to a temperature of about 60°C and is mixed well in a crutcher to form the hexahydrate salt of pentasodium tripolyphosphate. The preliminary crutcher mix thus made is then heated to 88°C and is maintained between that temperature and 93°C to prevent hydration of the anhydrous sodium tripolyphosphate powder to be added subsequently. The full crutcher mix is then made by addition, at a temperature in the mentioned 88° to 93°C range, of 28.3 parts of pentasodium tripolyphosphate powder (anhydrous) and 21 parts of deionized water. The mix resulting contains from about 45 to about 50% of solids by weight, due to hydration of some of the anhydrous tripolyphosphate and evaporation of some moisture.

The crutcher mix is pumped to a counter-current spray drying tower, which is 8 ft. high, and is sprayed at a manifold temperature of 82°C and at a pressure of about 750 p.s.i.g. (54 kg/cm² absolute) through a "Whirljet 15-1" spray nozzle into drying air having an initial temperature, as it enters the spray tower, of about 315°C.

The spray dried base beads produced are of internal structure and outer surface characteristics like those of the bead shown in Figures 6 and 7, being rounded solid particles of irregular configuration having sponge-like porous outer surfaces and skeletal internal structures, in contrast to conventional spray dried detergent beads which have a substantially continuous outer surface and a hollow core.

The spray dried base beads contain 77%

of sodium tripolyphosphate, 13% of sodium silicate and 10% of moisture. The bulk density is 0.55 g/cc, the flowability is 86% of that of dry sand and the product is completely non-tacky. A sieve analysis shows: 1% on a No. 20 screen; 19% through No. 20, on No. 40; 50% through No. 40, on No. 60; 20% through No. 60, on No. 80; 6% through No. 80, on No. 100; 3% through No. 100, on No. 200; and 1% through No. 200.

The base beads are introduced into a batch rotary drum blender and are post-sprayed at 49°C with "Neodol 25-7" and minor proportions of colouring agent, perfume and brighteners to produce a final product consisting of 78% of the base bead, 19.7% of "Neodol 25-7" and 2.3% of the minor components. In other experiments the liquids (the "Neodol 25-7" and the minor components or aqueous solutions or dispersions of them) are sprayed in the forms of fine droplets or mists onto the tumbling base beads in "Patterson-Kelley" twin shell and "Zig-Zag" blenders.

The compositions resulting are of a bulk density of 0.68 g/cc and a flowability of 79% and are completely non-tacky. They analyze: 1% on a No. 20 screen; 20% on No. 40; 52% on No. 60; 20% on No. 80; 5% on No. 100; 2% on No. 200; and 0% through No. 200.

The finished composition, obtained after only ten minutes of mixing, is filled at room temperature into bottles of the type illustrated in Figures 1-4 in the manner shown in Figure 8. The bottles are clear polyvinyl chloride bottles and hold a volume of $\frac{1}{2}$ gallon (approximately two litres). They are gravity filled with the detergent composition without incident, the average filling time being about five seconds per bottle or less and with the hollow handle being filled too, without any problems being encountered. After filling, the bottles are mechanically capped and the products made are packed in cases and sent to storage. Based on past experience with storage of heavy duty detergent compositions and tests run thereon it is considered that the products made have a storage stability in excess of three years.

Sufficient detergent composition to result in a concentration of 0.075% in a 17 gallon wash tub is obtained in 75 cc of the detergent composition, which is measurable by a cap approximately 5 cm in diameter and 4 cm high or by two capsful 4 cm in diameter and 3 cm high. The former cap size is employed with the bottle illustrated and described herein.

When the detergent composition is used in a top loading washing machine of 17 gallons tub capacity to wash an ordinary load of laundry (approximately 9 pounds) good washing is obtained. Similarly, when half as much is used with a front loading washing machine the laundry is effectively

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cleaned. During use it is noted that the product flows freely out of the bottle and out of the handle hollow, does not spill easily, as in the case of liquids, and does not leave undesirable gel or cement-like coatings on the bottle cap or threads.

The detergent composition is visually attractive and lends itself to identification colouring of some or all of the particles therein by known means, e.g. post-spraying some particles with dye.

Instead of making the detergent composition particles according to the foregoing described method, compositions of the same bulk density, particle size and flowability, which are also non-tacky, can be made by spray cooling, agglomeration and abrasion techniques, known in the art for manufacturing particulate detergent compositions, and useful bottled particulate detergent compositions result having the desirable detergency properties mentioned. However, those made by the method described earlier in this Example are superior to the products made by such other techniques, usually being more attractive, freer flowing and of greater bulk density for compositions of similar constitutions.

EXAMPLE 2

A product similar to that of Example 1 is made by essentially the same method, utilizing a crutcher premix of 25 parts of hot water (60°C), 3.5 parts of sodium silicate solids and 13 parts of pentasodium tripolyphosphate powder (anhydrous) and mixing the slurry well in a stream jacketed vessel to hydrate the phosphate to the hexahydrate and then heating it to 93°C with steam, at which temperature there is subsequently added to the crutcher mix 13 parts of the anhydrous tripolyphosphate, 25 parts of water, 13 more parts of anhydrous tripolyphosphate and 7.5 parts of anhydrous sodium carbonate. During the mixing the temperature is not allowed to fall below 82°C, to prevent hydration of the subsequently added anhydrous tripolyphosphate. The mix is sprayed at a pressure of 800 p.s.i.g. (57 kg/cm² absolute) into a spray tower having an inlet drying air temperature of 343°C and an outlet air temperature of about 113°C. The builder particles made are of a particle size distribution such that 90% by weight passes through a No. 20 screen and 90% by weight is retained on a No. 200 screen. 78 parts of the spray dried beads are then oversprayed by the method described in Example 1 with 19.5 parts of "Neodol 25-7" and 2.5 parts of minor ingredients (optical brighteners and perfume) as they are tumbled in an inclined cylindrical tumbling device for about five minutes. The composition removed has a bulk density of about 0.75 g/cm, a flowability rating of 75% and a moisture content of

about 5%. It is filled into the described bottles by the method of Example 1 and is tested for suitability as a heavy duty detergent composition by the method described therein. It is found to be very satisfactory, having the desirable properties previously reported for the Example 1 composition which was made by a similar method.

EXAMPLE 3

The procedures of Examples 1 and 2 are followed in making a base bead from: 13 parts of sodium tripolyphosphate hexahydrate; 26 parts of sodium tripolyphosphate, anhydrous; 47 parts of deionized water; 7.5 parts of organic builder "M" (Monsanto Chemical Company, U.S.A.); and 6.5 parts of sodium silicate solids (Na₂O:SiO₂=1:2.4). 85 parts of the spray dried builder beads resulting are oversprayed with "Neodol 45-11" (12 parts) and minor ingredients (3 parts of a total of fluorescent brighteners and perfume). The resulting detergent composition passes the tests mentioned in Examples 1 and 2 and is a free-flowing, dust-free, visually attractive, high bulk density laundry detergent composition of good detergency.

EXAMPLE 4

The experiment of Example 1 is repeated using "Alfonic 1618-65" as the nonionic detergent to provide a final granular detergent composition having a nonionic detergent content of 30%, with the proportions of other components being reduced correspondingly. The composition obtained has excellent detergency and the bottled particulate detergent composition is of excellent stability and use characteristics, as previously mentioned.

EXAMPLE 5

The experiments of Examples 1-4 are repeated with different bottles and caps and with different particulate detergent composition contents in them, as described previously, varying the proportions of the composition components $\pm 10\%$, $\pm 20\%$ and $\pm 30\%$ and maintaining them within the maximum ranges of proportions and ratios given herein, and filling them into different bottles, with caps, of different materials and structures, within the limits so given. Such compositions are readily made, free flowing, of high bulk density and readily measurable by the caps employed, and the detergent compositions contained therein are visually attractive and useful. Detergent compositions made by other manufacturing techniques, such as those described, which result in the same detergent particle characteristics, are similarly satisfactory.

The present invention avoids the problems associated with the use of liquids and for the first time allows the commercially acceptable use of particulate heavy duty detergent

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composition powders in a manner similar to that in which heavy duty liquid detergent compositions have been used, but without the disadvantages of such liquids. The bottles used are moisture-proof, promoting storage stability. They are attractive in appearance, relatively small, rescalable, easy to handle, and yet they allow the use of powdered detergent compositions where previously only liquids could be employed, with the disadvantages inherent in liquids.

WHAT WE CLAIM IS:—

1. A bottled particulate detergent composition which comprises a bottle having a neck for dispensing detergent composition from the bottle, and a free-flowing particulate heavy duty detergent composition in the bottle, pourable through the neck, the particles of which comprise synthetic organic detergent and builder for the detergent, are of particle sizes such that at least 90% thereof passes through an 8 mesh screen and is retained on a 200 mesh screen, are of a bulk density of at least 0.5 g/cc and are of a flowability at least 70% of that of clean dry sand.
2. A bottled particulate detergent composition according to Claim 1 wherein the neck of the bottle is at the top of the bottle and the passageway of the neck is of a cross-sectional area less than 40% of the average cross-sectional area of the volume of the bottle, the synthetic organic detergent of the particulate detergent composition is selected from anionic and nonionic detergents and mixtures thereof, the builder includes inorganic builder material and the particle sizes are such that at least 90% of the detergent composition passes through a 20 mesh screen and is retained on a 200 mesh screen.
3. A bottled particulate detergent composition according to Claim 1 or Claim 2 wherein the cross-sectional area of the bottle neck passageway is in the range from 2 to 40 cm².
4. A bottled particulate detergent composition according to any of Claims 1 to 3 wherein the bottle has an integral hollow handle the interior of which communicates with other interior portions of the bottle, the hollow handle including a portion of internal cross-sectional area in the range from 1 to 10 cm².
5. A bottled particulate detergent composition according to Claim 3 and Claim 4 wherein the cross-sectional area of the bottle neck passageway is in the range from 3 to 20 cm² and the internal cross-sectional area of the hollow handle portion is in the range from 2 to 5 cm².
6. A bottled particulate detergent composition according to any of Claims 1 to 5 wherein the bottle is of substantially oval cross-sectional shape and the neck is circular

and located on a major horizontal axis of the oval and nearer to one end of that axis than the other.

7. A bottled particulate detergent composition according to Claim 4 or Claim 5, and in Claim 6, wherein the neck is adjacent one side of the bottle near the end of the major horizontal axis of the bottle, and the handle is located at the other end of that axis.

8. A bottled particulate detergent composition according to any of Claims 1 to 7 wherein the neck is threaded and is covered by a removable screw cap of such size as to measure, when full, about the desired charge, about $\frac{1}{2}$ said charge, about $\frac{1}{3}$ said charge or about $\frac{1}{4}$ said charge, of the particulate detergent composition for use in washing one load of laundry in a washing machine.

9. A bottled particulate detergent composition according to any of the Claims 1 to 8 wherein the bottle is transparent and the detergent composition is dust-free so that, after pouring some of the detergent composition from the bottle, the bottle and the container volume above the level of the detergent composition remaining in the bottle are transparent, with no dust in the said volume and none clinging to the bottle interior wall above the said level of particulate detergent composition.

10. A bottled particulate detergent composition according to any of Claims 1 to 9 wherein the detergent composition includes phosphate and silicate builder salts.

11. A bottled particulate detergent composition according to any of Claims 1 to 10 wherein the detergent composition comprises from 60 to 98% of base beads of detergent builder having porous outer surfaces and skeletal internal structures and from 2 to 40% of a synthetic organic detergent material which is liquid or tacky at a temperature below 40°C, the synthetic organic detergent being disposed internally within the beads so that the outer surfaces of the beads are substantially free of the detergent.

12. A bottled particulate detergent composition according to Claim 11 comprising 70 to 95% of base beads and 5 to 30% of synthetic organic detergent, the base beads comprising from 45 to 85% of a phosphate builder salt, from 5 to 15% of alkali metal silicate and from 5 to 15% of water, and the synthetic organic detergent being a nonionic polyethoxylated detergent.

13. A bottled particulate detergent composition according to Claim 12 wherein the phosphate builder salt is sodium triphosphate, the alkali metal silicate is sodium silicate, the nonionic synthetic organic detergent is an ethoxylated aliphatic alcohol having a carbon chain of from 8 to 22 carbon atoms and from 5 to 30 ethylene oxide units per molecule, and the phosphate builder salt is present as a mixture of hydrated and



composition powders in a manner similar to that in which heavy duty liquid detergent compositions have been used, but without the disadvantages of such liquids. The bottles used are moisture-proof, promoting storage stability. They are attractive in appearance, relatively small, resealable, easy to handle, and yet they allow the use of powdered detergent compositions where previously only liquids could be employed, with the disadvantages inherent in liquids.

WHAT WE CLAIM IS:—

1. A bottled particulate detergent composition which comprises a bottle having a neck for dispensing detergent composition from the bottle, and a free-flowing particulate heavy duty detergent composition in the bottle, pourable through the neck, the particles of which comprise synthetic organic detergent and builder for the detergent, are of particle sizes such that at least 90% thereof passes through an 8 mesh screen and is retained on a 200 mesh screen, are of a bulk density of at least 0.5 g/cc and are of a flowability at least 70% of that of clean dry sand.

2. A bottled particulate detergent composition according to Claim 1 wherein the neck of the bottle is at the top of the bottle and the passageway of the neck is of a cross-sectional area less than 40% of the average cross-sectional area of the volume of the bottle, the synthetic organic detergent of the particulate detergent composition is selected from anionic and nonionic detergents and mixtures thereof, the builder includes inorganic builder material and the particle sizes are such that at least 90% of the detergent composition passes through a 20 mesh screen and is retained on a 200 mesh screen.

3. A bottled particulate detergent composition according to Claim 1 or Claim 2 wherein the cross-sectional area of the bottle neck passageway is in the range from 2 to 40 cm².

4. A bottled particulate detergent composition according to any of Claims 1 to 3 wherein the bottle has an integral hollow handle the interior of which communicates with other interior portions of the bottle, the hollow handle including a portion of internal cross-sectional area in the range from 1 to 10 cm².

5. A bottled particulate detergent composition according to Claim 3 and Claim 4 wherein the cross-sectional area of the bottle neck passageway is in the range from 3 to 20 cm² and the internal cross-sectional area of the hollow handle portion is in the range from 2 to 5 cm².

6. A bottled particulate detergent composition according to any of Claims 1 to 5 wherein the bottle is of substantially oval cross-sectional shape and the neck is circular

and located on a major horizontal axis of the oval and nearer to one end of that axis than the other.

7. A bottled particulate detergent composition according to Claim 4 or Claim 5, and in Claim 6, wherein the neck is adjacent one side of the bottle near the end of the major horizontal axis of the bottle, and the handle is located at the other end of that axis.

8. A bottled particulate detergent composition according to any of Claims 1 to 7 wherein the neck is threaded and is covered by a removable screw cap of such size as to measure, when full, about the desired charge, about $\frac{1}{2}$ said charge, about $\frac{1}{3}$ said charge or about $\frac{1}{4}$ said charge, of the particulate detergent composition for use in washing one load of laundry in a washing machine.

9. A bottled particulate detergent composition according to any of the Claims 1 to 8 wherein the bottle is transparent and the detergent composition is dust-free so that, after pouring some of the detergent composition from the bottle, the bottle and the container volume above the level of the detergent composition remaining in the bottle are transparent, with no dust in the said volume and none clinging to the bottle interior wall above the said level of particulate detergent composition.

10. A bottled particulate detergent composition according to any of Claims 1 to 9 wherein the detergent composition includes phosphate and silicate builder salts.

11. A bottled particulate detergent composition according to any of Claims 1 to 10 wherein the detergent composition comprises from 60 to 98% of base beads of detergent builder having porous outer surfaces and skeletal internal structures and from 2 to 40% of a synthetic organic detergent material which is liquid or tacky at a temperature below 40°C, the synthetic organic detergent being disposed internally within the beads so that the outer surfaces of the beads are substantially free of the detergent.

12. A bottled particulate detergent composition according to Claim 11 comprising 70 to 95% of base beads and 5 to 30% of synthetic organic detergent, the base beads comprising from 45 to 85% of a phosphate builder salt, from 5 to 15% of alkali metal silicate and from 5 to 15% of water, and the synthetic organic detergent being a nonionic polyethoxylated detergent.

13. A bottled particulate detergent composition according to Claim 12 wherein the phosphate builder salt is sodium tripolyphosphate, the alkali metal silicate is sodium silicate, the nonionic synthetic organic detergent is an ethoxylated aliphatic alcohol having a carbon chain of from 8 to 22 carbon atoms and from 5 to 30 ethylene oxide units per molecule, and the phosphate builder salt is present as a mixture of hydrated and



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and anhydrous salts, the ratio of hydrated phosphate to anhydrous phosphate being in the range from 0.3:1 to 0.7:1.

14. A bottled particulate detergent composition according to Claim 13 wherein the detergent composition is of particle sizes over 95% of which are in the range from 40 mesh to 200 mesh, with less than 0.5% passing through a 200 mesh sieve, the bulk density of the detergent composition particles is in the range from 0.55 to 0.8, and the flowability of the detergent composition is at least 75% of that of clean dry sand.

15. A bottled particulate detergent composition according to Claim 14 wherein the sodium silicate of the synthetic organic detergent composition is of $\text{Na}_2\text{O}:\text{SiO}_2$ ratio in the range from 1:2 to 1:3 and of particle sizes such that the percentage passing through a 200 mesh sieve is nil, and the nonionic detergent is a condensation product of an aliphatic alcohol having a carbon chain of 10 to 18 carbon atoms and 6 to 14 mols of ethylene oxide per mol of the aliphatic alcohol.

16. A bottled particulate detergent composition according to any of the preceding Claims wherein the bottle is made of a synthetic organic polymeric plastics material or glass.

17. A bottled particulate detergent composition according to Claim 16 wherein the bottle is made of polyvinyl chloride.

18. A bottled particulate detergent composition substantially as described with reference to Figures 1 to 4 of the accompanying drawings.

19. A bottled particulate detergent composition substantially as described in any of the Examples.

20. A method of filling with a particulate detergent composition a bottle having a hollow handle communicating with a main volume of the bottle without special filling

operations to make sure that the hollow 45
handle section is filled with particulate
detergent composition, which method com-
prises maintaining in an upright position
a bottle of elongate horizontal cross-section 50
having long and short major and minor
horizontal axes, the long axis defining a
vertical plane and the short axis defining
an intersecting vertical plane, with a neck
at the top of the bottle near one end of the
vertical plane defined by the long horizontal 55
axis and an integral hollow handle at the
other end of this vertical plane, the hollow
of which handle communicates with the main
volume of the bottle, and directing particulate
detergent composition downwardly through 60
the bottle neck to the main volume of the
bottle to fill the main volume of the bottle
and the handle thereof, the filling of the
handle being effected by flow of the particu- 65
late detergent composition downwardly into
the handle when the detergent composition
level rises above the handle, without tilting
the bottle or directing particulate detergent
into the handle, the bottle neck passageway
being of a cross-sectional area in the range 70
from 3 to 20 cm^2 and less than 40% of the
average cross-sectional area of the bottle,
the hollow handle including a portion of
internal cross-sectional area in the range 75
from 2 to 5 cm^2 , and the particulate detergent
composition being of particle sizes such that
at least 90% thereof passes through a 20
mesh screen and is retained on a 200 mesh
screen, the composition being of a bulk 80
density of at least 0.5 g/cc and of a flow-
ability at least 70% of that of clean dry sand.
21. A method according to Claim 20,
substantially as described with reference
to Figure 8 of the accompanying drawings.

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Chartered Patent Agents,
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Printed for Her Majesty's Stationery Office by Burgess & Son (Abingdon), Ltd.—1981.
Published at The Patent Office, 25 Southampton Buildings, London, WC2A 1AY,
from which copies may be obtained.