METHOD OF PREPARING AGGLOMERATED DETERGENT COMPOSITION

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FIG. 1

FIG. 2

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A method of producing agglomerates of dry detergent ingredients by charging particulate detergent ingredients into an agglomeration zone and maintaining a continuously falling curtain of said ingredients in said zone, contacting said particulate material in said falling curtain with a sodium salt of an alkyl aryl sulfonic acid to agglomerate said particulate material, maintaining a tumbling bed of agglomerating ingredients at the base of said falling curtain and withdrawing agglomerated material from said agglomeration zone, said sodium salt of alkyl aryl sulfonic acid being prepared in situ by merging sprays of liquid caustic soda and alkyl aryl sulfonic acid.

This invention relates to a method of producing a detergent composition. More particularly, it relates to a process utilizing known detergent composition ingredients such as, e.g., laundry detergent ingredients for producing agglomerates of these ingredients, such process having several advantages over prior art processes. One such advantage is that it is more economical than prior art processes.

Another advantage is that laundry detergents having characteristics similar to those obtained by well-known spray-dried processes can be obtained without several of the disadvantages of such processes.

The vast majority of laundry detergent powders are manufactured by the spray-drying process, because spray-dried powders have many advantages over the other types. These advantages can be summarized: (a) The formulation is not limited. Relatively high amounts of active matter can be incorporated; soda ash is not an essential ingredient, and the moisture and bulk density can be varied at will (within definite limits). (b) The powders present a pleasing appearance and being light have more sales appeal. (c) Spray-dried powders are dustless and free-flowing and do not tend to lump. For normal formulations, no special inner liners are required in packaging. (d) Because it consists of hollow beads, with a large surface area, the powder dissolves instantly when added to water. This is important when powders are used in machines where the wash cycle can be as low as 4 minutes. If the powder takes time to dissolve, valuable washing time is lost. Also, in tub-washing, where no mechanical agitation takes place, portions of powders other than spray-dried powders, can still be undissolved even when the operation has been completed. (e) Heat-sensitive materials can be safely handled in a spray-drier.

Many types of spray-driers are available, both of the jet and of the disc type, and also using both counter-current and co-current airflow patterns. Most, although not all, spray-driers for detergent powders are of the counter-current airflow, jet-spray type, which produce a large bead with a minimum of dust and a medium bulk density. However, spray-drying processes for preparing laundry detergents have the disadvantage, among others, that the spray-drier needed in the process involves a large capital outlay.

The other principal process for preparing laundry detergent powders involves the absorption of a surfactant onto inorganic salts in conventional beaded powder mixers such as ribbon mixers, plough mixers, incorporators, or screw mixers.

This method of producing powders is to charge the mixer with the dry ingredients, start the mixing, and to pump (or pour) a liquid surfactant through a jet into the powder slowly while mixing. The rate of addition of the liquid surfactant must be adjusted so that the liquid is absorbed as it reaches the surface of the powder; if not, hard crystalline lumps will be formed.

After all the liquid has been added, mixing is continued for at least 15 minutes. In general, the aging serves the purpose of allowing the crystals to form and cool.

In this method, a free-flowing powder, free from lumps, is desired and generally, is obtained; but if any of the original dry ingredients contained an appreciable amount of lumps, it will be necessary to pass the powder through a hammer mill after aging.

This process permits only a limited amount of active matter to be incorporated into the powder, and it yields materials with only limited application for household use.

The laundry detergent composition prepared by the process of this invention is free-flowing, non-caking and non-friable. Laundry detergents having characteristics similar to those obtained with the spray-dried process, are obtained without the expense of the spray-drier. Several of the limitations of processes involving absorption are overcome.

A principal object of the present invention is to provide a process for the preparation of detergent composition.

An object of the present invention is to prepare by a unique process involving agglomeration the well-known spray-dried detergents.

Still another object of this invention is to prepare a homogeneous, non-segregating substantially dust-free agglomerate of laundry detergent ingredients in either a batch or continuous operation.

A further object is to provide a process for preparing laundry detergent compositions wherein it is possible in one operation to mix dry particulate ingredients, and then add a sodium salt of alkyl aryl sulfonic acid, as hereinbefore defined, prepared in situ by merging sprays of liquid caustic soda, as hereinafter defined, and an alkyl aryl sulfonic acid and run on a batch or continuous basis to produce a laundry detergent composition.

These and other objects are accomplished in accordance with the present invention in the following manner. Generally, in a continuous operation, dry laundry detergent composition ingredients, in particulate form such as in powder or fine crystalline form, are charged into a bed of material being agglomerated in a suitable agglomeration zone. A preferred agglomeration zone comprises an elongated cylindrical confined zone which is rotated about its longitudinal axis and which is adapted with suitable means for maintaining a continuously falling curtain of the detergent ingredients communicating between an upper portion of the confined zone and a bed of the material maintained in the bottom portion thereof.

A sodium salt of alkyl aryl sulfonic acid, defined hereinafter, as discrete droplets, is brought into contact with the particulate ingredients in the falling curtain, preferably of a constant density, thereby wetting the particles to form agglomerates, preferably without the sodium salt of alkyl aryl sulfonic acid, contacting any part of the apparatus being used. The agglomerated particles fall into the bed of material maintained in the bottom portion of the agglomeration zone where they are subjected to shear which breaks the few large oversize agglomerates that happen to form into desirable uniform-sized particles. The rotating and tumbling action of the bed produces
agglomerated particles of the laundry detergent composition of the desired size. Particle size of the agglomerates can be varied by controlling the rate of rotation of the drum, the length of time of rotation, and the amount of the sodium salt of alkyl aryl sulfinic acid applied. The discrete droplets of the sodium salt of alkyl aryl sulfinic acid are prepared in situ in the said agglomeration zone by merging sprays of liquid caustic soda, hereinafter defined, and alkyl aryl sulfinic acid, hereinafter defined, both materials having been sprayed as discrete droplets through suitable spray nozzles at the proper angle.

More specifically referring to the drawings, with particular initial reference to FIG. 1, the apparatus may generally comprise a drum 16, comprising an outer cylindrical shell or wall 28; an annular end plate 32 defining a feed opening 34 through which the particular ingredients 36 are loaded; an annular end plate 38 defining a discharge opening 40 through which the agglomerated detergent composition 44 is discharged through hopper 42; a spiral conveyor vane 46 to move the particular ingredients and/or partially agglomerated particle 36 through the drum toward the feed opening 34; and a spiral conveyor vane 46 to move the particular ingredients and/or agglomerated particle 44 toward the discharge opening 40; a feed line 56 provided with a plurality of spaced spray nozzles 58 (FIGS. 2 and 3); a second feed line 57 provided with a plurality of spaced spray nozzles 57 and a cylindrical bundle of parallel-spaced cylindrical rods 60 which develop the continuously falling curtain and are carried between conveyor vanes 46 and 52 and positioned between drum end plates 32 and 38. It will be appreciated that, except for feed line 56 and 57, the components of drum 16 rotate together as an integral unit. Nozzles 58 and 59 should be positioned so that the material sprayed from them, as hereinafter specified, intersect each other between nozzles 58 and 59 and said falling curtain. The angle of the intersection of said sprays from nozzles 58 and 59 is not critical but it should be as small as possible to allow maximum intimate contact of the two sprays, thus giving maximum reaction time, and should not be so great as to prevent the reaction product of the two particular materials being sprayed from being carried onto said falling curtains.

Other apparatus that could be modified to be useful in the practice of this invention is described in pending application Ser. No. 858,213, filed May 23, 1969, to which reference is hereby made.

The ingredients that are useful in preparing laundry detergent composition are well-known to those skilled in the art. They have been described in numerous patents and articles.

Specifically, the ingredients for the detergent composition, especially laundry detergent composition, that can be made by the process of this invention include the following: (a) alkaline condensed phosphate, (b) sodium or potassium silicates, (c) neutral soluble salts, (d) sodium salt of alkyl aryl sulfinic acid, and optionally, (e) alkalies, and (f) such materials as insoluble inorganic builders and special purpose additives.

The alkaline condensed phosphates preferably are those having an Na₂O or K₂O to P₂O₅ ratio of about 1:1 to 2:1. More preferably, they are the pyrophosphates and polyphosphates, the more desirable are those that rapidly hydrate. The most preferred alkaline condensed phosphate is sodium tripolyphosphate. About 15 to 70 parts by weight of the alkaline condensed phosphate per 100 parts is useful in the composition, preferably the amount is about 30 to 60 parts by weight. The alkaline condensed phosphates can be used in anhydrous form or in a hydrated or partly hydrated form, preferably the anhydrous form.

The sodium or potassium silicates preferably are those having an Na₂O or K₂O to SiO₂ ratio of about 1:3.75 to 2:1. More preferred, are the sodium silicates having an Na₂O to SiO₂ ratio of about 1:2.2 to 1:2.5. Preferably, about 5 to 20 parts by weight silicate (on an anhydrous basis) per 100 parts detergent should be used. A more preferred ratio is about 1 to 10. The silicate can be used in its anhydrous form or in its hydrated form or as a water solution of these forms or a combination thereof.

Water solutions of silicates are known as liquid silicates and normally are the sodium silicates having an Na₂O:SiO₂ ratios ranging from about 1:1.60 to about 1:3.75.

"Liquid" caustic soda is (1) a solution of caustic soda (NaOH) in a suitable solvent, preferably water, which solution is maintained in a liquid state at ambient temperatures by heating if necessary or (2) 100% caustic soda maintained in a liquid state by heating it to above its melting point.

Preferably the caustic soda should be about a 40% or greater aqueous solution or 100% caustic soda, more preferably, the caustic soda should be about a 75% or greater aqueous solution or 100% caustic soda. Most preferably, it should be about a 75% aqueous solution.

Thus, the concentration of caustic soda, which can be employed in the practice of the present invention, includes the readily available concentrations of about 50 and 73% NaOH in aqueous solution.

If the aqueous solution of caustic soda is in a solid state at ambient temperature, it will be necessary to heat in order to liquify it before spraying it as previously described. Such is also true of 100% caustic soda; however, here it must be melted to a molten state.

The sodium salts of the so-called "alkyl aryl sulfinic acids" that are useful in the practice of this invention are well-known by those skilled in the art. This material is the reaction product of liquid caustic soda and alkyl aryl sulfinic acid, both hereinafter defined. About 8 to 30 parts by weight of said sodium salt per 100 parts detergent is useful in the practice of this invention. It is preferred to use 15 to 25 parts by weight per 100 parts detergent.

As previously stated, the salt is prepared in situ in the agglomeration zone useful in the practice of this invention by simultaneously spraying liquid caustic soda, herefore defined, and alkyl aryl sulfinic acid, herefore described, in the manner heretofore described.

The alkyl aryl sulfinic acids that are useful are a recognized group of chemical compounds and are well-known by those skilled in the art. Most of these compounds are the sulfonation product of an alkylated aromatic nucleus, which product can be neutralized with caustic soda to prepare the class of anionic surface-active agents known as alkylarenesulfonates, or commonly, alkyl aryl sulfonates. These alkyl aryl sulfinic acids are described in numerous literature articles, for example, Kirk-Othmer Encyclopedia of Chemical Technology, vol. 13, "Sulfonation and Sulfation," pages 317-337, and "Surface-Active Agents," pages 513-523.

The preferred alkyl aryl sulfinic acids are those in which the alkyl group is straight or branched (most preferably straight) and contain 8 to 24 carbon atoms and the aryl group is benzene. The straight chained ones being the "biodegradable" type. Another preferred alkyl aryl sulfinic acid is the so-called alpha olefin sulfinic acid, particularly those taught in U.S. 3,392,874 through 3,392,880 incorporated herein by reference. The most preferred alkyl aryl sulfonic acid is dodecylbenzene sulfinic acid, wherein in the manufacture thereof, the petroleum fraction used to form the side chain is so chosen as to have an average molecular weight corresponding to twelve carbon atoms, but is, of course, a mixture.
The term "alkalies" includes sodium hydroxide, sodium carbonate, sodium bicarbonate, sodium sesquicarbonate, sodium borate (borax) or the potassium analogs thereof.

The term "neutral soluble salts" include sodium sulfate and sodium chloride. About 1 to 60 parts by weight of the neutral soluble salts per 100 parts detergent is useful. It is preferred to use 3 to 10 parts by weight of the neutral soluble salts per 100 parts of the detergent.

Preferably, the detergent products of the process of this invention contain an alkali condensed phosphate, sodium or potassium silicates, neutral soluble salts, the surfactant and one or more optional ingredients, including alkalies.

Various optional ingredients can also be included for various uses purposes. These materials include insoluble inorganic builders, such as clays; various organic builders and special purpose additives, such as sodium carboxymethyl cellulose; heavy metal sequestering agents; foam builders and stabilizers; solubilizing additives; optical bleach or brightening agents; corrosion inhibitors; tarnish inhibitors, and others, including enzymes.

Specifically excluded from the detergent compositions that can be prepared by the process of this invention are active chlorine containing compounds. These compounds are defined in U.S. application Ser. No. 764,634, filed Oct. 1, 1968, and are defined therein as follows:

The active chlorine-containing compound imparts germicidal, bleaching, water sheeting, and protein removing action to the detergent compositions. They include chlorinated trisodium phosphate, trichlorocynamuric acid, the sodium salt of dichlorocynamuric acid, the potassium salt of dichlorocynamuric acid, sodium hypochlorite and 1,3-dichloro-5,5-dimethyl hydantoin.

Normally it is preferred to sell detergents such as the "bulk blended detergents" as distinguished from the "spray-dried detergents" with all of the hydratable ingredients fully hydrated. Therefore, in the practice of this invention, if all hydratable ingredients such as the alkali condensed phosphates are not fully hydrated, it is preferred to hydrate them during the process of this invention.

The ingredients of the detergent compositions are combined as follows: According to one method, all the particulate ingredients are mixed to form a homogeneous mixture, if they are not already so mixed. Such particulate ingredients are the alkali condensed phosphates, and, if desired, sodium or potassium silicates, alkalies, neutral soluble salts, and optional particulate ingredients.

Next, the liquid caustic soda and the alkyl aryl sulfonic acids are simultaneously sprayed each from a separate system of nozzles in such a manner that the two sprays intersect each other, preferably at an acute angle, and the resulting spray mixture reaction product continues and is sprayed as discrete droplets into a falling curtain of the particulate ingredients.

Water can be sprayed either before, during, or after the spraying of the sodium salt of alkyl aryl sulfonic acid to hydrate the hydratable ingredients. Preferably the sodium salt of alkyl aryl sulfonic acid is added onto the particulate ingredients that are not fully hydrated.

Generally, the size of the agglomerates that can be produced in the practice of this invention is between about 10 and about 60 U.S. mesh size, preferably a mesh size between 12 and about 40 is produced. Normally, the particle size of the powdered dry ingredient used in preparing the agglomerates is about 100 U.S. mesh size.

Having thus described the invention in general terms, reference is now made to the figure of the drawing which describes one preferred apparatus for carrying out the process of the present invention. The preferred apparatus is described herein and illustrated in the figure of the drawing.

The following examples will serve to illustrate the process of this invention. The amounts of the ingredients are expressed in kilograms, unless otherwise indicated.

**EXAMPLE I**

A representative agglomeration is carried out batch-wise in the agglomerator apparatus, similar to that shown in the drawings and described heretofore using the following recipe:

1. (1) Particulate ingredients:
   - Sodium tripolyphosphate 100
   - Soda ash 68
   - Carboxymethyl cellulose and dye 1.3
   - Sodium sulfate 68
   - Liquid caustic soda, 40% by weight 25
   - Dodecyl benzene sulfonic acid 75
   - Liquid sodium silicate 44% by weight 34

First, the dry particulate ingredients are added and allowed to dry blend to a homogeneous mixture, usually for about 2 minutes. Next, the reaction product of a mixture of caustic soda and the acid is sprayed onto a falling curtain of the mixed particulate ingredients, the caustic soda having been sprayed from nozzles at a pressure of about 30 to 60 p.s.i. at a temperature of 80-100° F., and the acid having been sprayed from nozzles at a pressure of about 60 to 100 p.s.i. at a temperature of 100 to 180° F., the sprays having been merged as shown in FIG. 3, said spraying being for about 5 to 10 minutes. Finally, the liquid sodium silicate is sprayed at a temperature of about 180° F. at a pressure of about 80 p.s.i. for about 10 minutes.

The rotation is continued for about 20-30 minutes to cool the resulting agglomerates.

I claim:

1. A method of producing agglomerates of detergent ingredients which comprises (1) charging dry particulate detergent ingredients consisting essentially of (A) about 20 to 80 parts by weight of an alkali condensed phosphates having a Na₂O or K₂O to P₂O₅ ratio of about 1:1 to 2:1, (B) about 5 to 20 parts by weight sodium or potassium silicate (on an anhydrous basis) having an Na₂O or K₂O to SiO₂ ratio of about 1:3.75 to 2:1, (C) about 1 to 60 parts by weight of a neutral soluble salt all per 100 parts detergent into an agglomeration zone, (2) maintaining a continuously falling curtain of said ingredients in said zone (3) contacting said ingredients in said falling curtain with about 8 to 30 parts by weight per 100 parts detergent a sodium salt of alkyl benzene sulfonic acid to agglomerate said particulate material, said sodium salt of alkyl benzene sulfonic acid, said alkyl group having 10 to 18 carbon atoms, being prepared in situ in said agglomeration zone by merging sprays of liquid caustic soda and alkyl sulfonic acid, said alkyl group having 10 to 18 carbon atoms, (4) maintaining a tumbling bed of agglomerating detergent ingredients at the base of said falling curtain, and (5) withdrawing agglomerated material from said agglomeration zone.

2. The method of claim 1 in which said withdrawing agglomerated material from said agglomeration zone is done continuously.

3. The method of claim 1 in which said alkyl benzene sulfonic acid is an alkyl benzene sulfonic acid in which said alkyl group is linear and has from 10 to 18 carbon atoms.

4. The method of claim 3 in which said alkyl benzene sulfonic acid is dodecyl benzene sulfonic acid.

5. The method of claim 3 in which said caustic soda is about 40% or greater aqueous solution.

6. The method of claim 3 in which said caustic soda is about 73% or greater aqueous solution.

7. The method of claim 3 in which said caustic soda is about 73% aqueous solution.

8. The method of claim 4 in which said caustic soda is about 73% aqueous solution.

9. The method of claim 1 together with the additional step of contacting said ingredients in said falling curtain with liquid sodium silicate.
10. The method of claim 3 in which said caustic soda is about 100% caustic soda.

11. The method of claim 1 in which said particulate detergent ingredients consist essentially of (A) about 30 to 60 parts by weight sodium tripolyphosphate (B) 5 to 20 parts by weight sodium silicate (on an anhydrous basis) having an Na₂O to SiO₂ ratio of about 1:3.22 to 1:2.5 (C) about 3 to 10 parts by weight of a neutral soluble salt, all per 100 parts detergent and 15 to 25 parts by weight per 100 parts detergent of said sodium salt of alkyl benzene sulfonic acid is used to contact said particulate ingredients.

12. The method of claim 1 in which said detergent ingredients also include materials selected from the group consisting of alkalies, insoluble inorganic builders and special purpose additives.

13. The method of claim 11 in which said detergent ingredients also include materials selected from the group consisting of alkalies, insoluble inorganic builders and special purpose additives.