PROCESS FOR PREPARING A GRANULAR AUTOMATIC DISHWASHING DETERGENT COMPOSITION

Inventors: Gary W. Kingry; Frank H. Lahrman, both of Cincinnati, Ohio
Assignee: The Procter & Gamble Company, Cincinnati, Ohio
Filed: Sept. 5, 1972
Appl. No.: 286,654

U.S. Cl. 252/99; 252/135; 252/174
Int. Cl. C11d 7/54
Field of Search 252/99, 135, 174

References Cited
UNITED STATES PATENTS
3,598,743 8/1971 Coates 252/99
3,620,979 11/1971 Corliss et al. 252/135 X
3,630,928 12/1971 Fuchs 252/99 X
3,701,736 10/1972 Austin et al. 252/99
3,763,047 10/1973 Fairs 252/99

Primary Examiner—Benjamin R. Padgett
Assistant Examiner—E. A. Miller
Attorney, Agent, or Firm—Charles R. Wilson; Julius P. Filick; Richard C. Witte

ABSTRACT
A method of producing an automatic dishwasher detergent composition comprising the steps of charging a particulate alkaline builder salt to a mixture zone, agitating the particulate matter and spraying thereon a mixture of liquid silicate and nonionic detergent. The resultant composition is in the form of crisp, freeflowing agglomerated granules. In a preferred aspect, a high level of nonionic detergent is included in the formulation without destroying the crispness of the granular product.

10 Claims, No Drawings
PROCESS FOR PREPARING A GRANULAR AUTOMATIC DISHWASHING DETERGENT COMPOSITION

BACKGROUND OF THE INVENTION

The present invention relates to a process of preparing an automatic dishwasher detergent composition. In a preferred aspect, it relates to a process of producing a dishwasher detergent composition which is crisp and free flowing and contains a high level of nonionic detergent.

Automatic dishwasher detergent compositions generally contain an alkaline builder salt, a bleach, an alkali metal silicate and a small level of water-soluble organic synthetic nonionic detergent. The alkaline builder salt sequesters or ties up hardness ions normally found in tap water – thus allowing the therein included detergent to more satisfactorily perform its function. The purpose of the bleach component is to break up food particles into a form which is more easily dissolvable and, hence, more easily removable from tableware. An alkali metal silicate is included in the known compositions for its corrosion inhibiting effect. The generally included nonionic detergent performs a cleaning function as well as providing a “water sheeting” effect. The latter function is important in that it allows for water to more easily drain from tableware thus leaving the tableware with a spotless appearance. Such known compositions are generally found in the form of agglomerates. The dry components are normally admixed and thereafter sprayed with water or a liquid silicate. The spray on liquid material has the effect of causing the agglomeration of the small particulate matter into larger forms.

Preferably, a relatively high level of nonionic detergent is desired in a dishwasher detergent composition. Unfortunately, nonionic detergents when included in a granular detergent composition by prior art methods tend to make the total composition tacky and non-free flowing. For this reason, generally less than 3% by weight nonionic detergent is included in dishwasher detergent compositions. Various processes and additive materials have been used in an attempt to overcome the caking problem inherent with high levels of nonionic detergents in such compositions. While some prior art attempts have been successful to a limited degree, there is still a need to include higher levels of nonionic detergents in dishwashing detergent compositions without experiencing a caking effect.

An object of the present invention is the production of an automatic dishwasher detergent composition by an economical process.

Another object of this invention is to produce a non-caking automatic dishwasher detergent composition.

A still further object is to produce an automatic dishwasher detergent composition that contains a high level of nonionic detergent and is non-caking.

These and other objects will become apparent from the description and the examples to follow.

As used herein, all percentages are by weight unless otherwise specified.

SUMMARY OF THE INVENTION

A process for producing a crisp, free-flowing, agglomerated granular automatic dishwasher detergent composition comprising the steps of:

1. Charging to a mixing zone particulate matter comprising an alkaline builder salt;
2. Agitating the particulate matter in the zone; and
3. With continuous agitation spraying onto the particulate matter a liquid mixture of an alkaline metal silicate having an SiO₂:alkali metal oxide ratio of from 3.6:1 to 0.5:1 and a synthetic nonionic detergent wherein the ratio of liquid alkali metal silicate to nonionic detergent ranges from 2.5:1 to 20:1 and wherein the liquid mixture is sprayed on at a level of from 25% to 50% based on the total composition to form said agglomerated granules.

DETAILED DESCRIPTION OF THE INVENTION

The process of the present invention comprises initially charging particulate matter comprising an alkaline builder salt to a mixing zone. The particulate matter is then agitated and while being agitated has sprayed thereon a mixture of liquid silicate and a nonionic detergent. As a result of the spray-on of the liquid mixture, the particulate solids form agglomerates which are crisp and free flowing.

The alkaline builder salts that form a part of the composition of this invention are included therein in the range of from 10% to 70%, preferably 25% to 50%, based on the total final composition. The particular builder salts that form a part of this invention are any of the well-known sequestering type or precipitating type builder salts commonly used in automatic dishwasher detergent composition. Particularly preferred builder salts are sodium tripolyphosphate, alkali metal carbonates, and alkali metal citrates.

Additional particulate components that can be included with the alkaline builder salts include sodium sulfate, a chlorine-yielding bleach as more fully described hereinafter, various known suds suppressors, coloring matter and dyes.

The particulate matter may have a wide range of particle sizes. Preferably, all the particles will pass through a Tyler No. 8 screen.

As the first step of the process of this invention above, particulate matter is charged to a mixing zone. Any suitable mixing device such as an inclined pan granulator, a rotating drum, or any other vessel with suitable means of agitation may be used. Methods of agitating the particulate components of this invention are well known to those skilled in the art. With continuous agitation, a liquid alkali metal silicate and nonionic detergent mixture is sprayed onto the particulate mixture. The alkali metal silicate has an SiO₂:alkali metal oxide ratio of from 3.6:1 to 0.5:1, preferably 3.2:1 to 2.5:1. The liquid silicates used herein contain from 45% to 70%, preferably 55% to 65%, water.

The nonionic detergents found most useful in the instant detergent composition are the low-foming nonionic compounds. Compounds which meet the above criteria and which are advantageously employed in the composition of this invention include, but are not limited to, the following polyoxyalkylene nonionic detergents: C₆H₄(CH₂O)₇H where y equals at least 15 and (C₆H₄O)₂y equals 20–90% of the total weight of the compound; alkyl polyoxypropy-
llex-polyoxyethylene condensates having the formula $RO-(CHO)(CHO)H$ where $R$ is a $C_1-C_{12}$ alkyl group and $x$ and $y$ represent an integer from 2 to 98; polyoxyalkylene glycols having a plurality of alternating hydrophobic and hydrophilic polyoxyalkylene chains; the hydrophilic chains consisting of linked oxyethylene radicals and the hydrophobic chains consisting of linked oxypolypropylene radicals, said product having three hydrophobic chains, linked by two hydrophilic chains, the central hydrophobic chain consisting of 30% to 34% by weight of the product, the terminal hydrophobic chains together constituting 31% to 39% by weight of the product, the linking hydrophobic chains together constituting 31% to 35% by weight of the product, the intrinsic viscosity of the product being from 0.06 to 0.09 and the molecular weight being from about 3,000 to 5,000 (as described in U.S. Pat. No. 3,048,548); butylene oxide capped alcohol ethoxylates having the formula $R(OC\text{H}_2\text{H})_y(OC\text{H}_2\text{H})_x\text{OH}$ where $R$ is a $C_6-C_{20}$ alkyl group and $x$ is an integer from about 3.5 to 10 and $y$ is an integer from about 0.5 to 1.5; benzyl ethers of polyoxyethylene condensates of alkyl phenols having the formula \[
\begin{align*}
\text{R} & \quad (\text{OC}\text{H}_2\text{H})_x \quad \text{OCH}_2\text{C}_6\text{H}_5 \\
\end{align*}
\]
where $R$ is a $C_6-C_{20}$ alkyl group and $x$ is an integer from 3 to 20. Other nonionic detergents are suitable for use in the herein-disclosed dishwashing compositions and it is not intended to exclude any detergent possessing the desired attributes.

The amount of liquid mixture sprayed onto the particulate mixture is sufficient to cause the particulate mixture to form a bed of agglomerated granules but not so great that its particulate nature is destroyed. From 25% to 50%, preferably 35% to 45% of the liquid mixture based on the total composition wherein the ratio of liquid silicate to nonionic detergent is from 2.5:1 to 3.5:1, preferably 3:1 to 6:1, is sprayed onto the particulate mixture.

The liquid mixture is preferably agitated continuously prior to spray-on so as to prevent any separation of the alkali metal silicate and nonionic detergent. The temperature of the liquid mixture is maintained at a temperature of from 110°F. to 200°F., preferably 140°F. to 160°F. At these temperatures proper atomization is achieved as well as the formation of agglomerates having an optimum particle size.

Spraying of liquid silicate and nonionic detergent separately onto the dry particulate mixture does not give a product as crisp or free flowing as the product resulting from the process of this invention. It is not known exactly why the spray-on of the mixture in accord with this invention gives a more crisp, free-flowing product than does the separate spray-on of the same components at the same weight level. It is believed that the resultant product is crisp and free flowing due to the tacky nonionic detergent being enclosed within the silicate matrix as the agglomerated particles cool or hydrogen bonding existing between the nonionic detergent and silicate.

The agglomerated granules resulting from the above-described spray-on may next be subjected to a drying operation and thereupon a grinding and classification operation to achieve a product having substantial uniformity in particle size. Preferably the resultant final product has a particle size such that all the particles will pass through a Tyler No. 8 screen.

As a result of the process of this invention a level of nonionic detergent can be used in excess of that normally used without destroying the crispness and flow properties of the final product. As previously discussed, such is desirable from a performance standpoint. In this preferred aspect of the invention from 5% to 10% of the nonionic is added to the composition by way of the present process. The resultant product is crisp, free-flowing and performs very satisfactorily with respect to cleaning and spotting/filming.

The bleach component previously mentioned that may be a part of the particulate matter is a chlorine-yielding bleach. Such bleach is included in the composition at a level sufficient to give the detergent composition an available chlorine content of from 0.5% to 10%, preferably 1% to 5%. As used herein, the term "available chlorine" indicates the amount of chlorine in the composition which is equivalent to elemental chlorine in terms of oxidizing power. "Active chlorine" is oftentimes used instead of "available chlorine." The same type of chlorine is designated by the two terms, but when expressed quantitatively "active chlorine" indicates the chlorine actually present. The numerical value for available chlorine content is twice that for active chlorine. Available chlorine contents below 0.5% fail to give proper cleaning performance, while amounts in excess of 10% do not result in any added cleaning ability. Any of many known chlorine bleaches can be used in the present detergent composition. Examples of such bleach compounds are: chlorinated tri-sodium phosphate; dichlorocyanuric acid; salts of chlorine substituted cyanuric acid; 1,3-dichloro-5,5-dimethylhydantoin; N,N'-dichlorobenzoylene urea; paratoluene sulfonic dichlorohemiamide; trichlororolameline; N-chloroanilidene; N-chlorosuccinimide; N,N'-dichlorooazodicarbonamide; N-chloroacetetyl urea; N,N'- dichloroblueter; chlorinated dicyandiamide; sodium hypochlorite; calcium hypochlorite; and lithium hypochlorite. The preferred bleach is an alkali metal salt of dichlorocyanuric acid, e.g. potassium or sodium dichlorocyanurate. Depending on the particular bleach utilized, the bleach may be included with the particulate alkaline builder salt prior to the liquid mixture spray-on or may be admixed with the agglomerated granules of alkaline builder salt, nonionic detergent and silicate. That is, a bleach that is susceptible to high levels of water and/or heat must be admixed with the agglomerated granules. Similarly a bleach that is not susceptible to water or heat degradation but is of a particle size smaller than desired in the final product must be included with the particulate alkaline builder salt.

The following examples are illustrative of the invention.

**EXAMPLE I**

A particulate mixture 55 parts by weight of sodium citrate dihydrate and 15 parts by weight of sodium carbonate was charged to a pan agglomerater. The particle
size of this mixture was such that all the particles passed through a Tyler No. 8 screen. A liquid mixture of 20.5 parts by weight of liquid sodium silicate having a SiO$_2$:Na$_2$O ratio of 2.58:1, 20.5 parts by weight of liquid sodium silicate having a SiO$_2$:Na$_2$O ratio of 3.20:1, and 9.5 parts by weight of a polyoxyalkylene nonionic detergent (Pluradot HA-433 nonionic detergent supplied by the Wyandotte Corp.) was prepared. The liquid mixture contained 50% water. The liquid mixture was agitated and heated to a temperature of 140°F. As the citrate and carbonate mixture was being agitated, the liquid mixture of sodium silicate and nonionic detergent was sprayed thereon. Agglomeration of the particulate mixture began as the spray droplets contacted it. After completion of the spray-on, the agglomerated particles were dried to a moisture content of 10%. The agglomerates were thereafter ground and classified so that all the agglomerates passed through a Tyler No. 8 screen and more than 10% passed through a Tyler No. 100 screen.

For purposes of comparison, products having the above formulation were prepared by different processes. In one process, the liquid sodium silicate was first sprayed onto the particulate mixture, followed by the spray-on of the nonionic detergent. In another process, the nonionic detergent was first sprayed onto the particulate mixture, followed by the spray-on of the liquid sodium silicate. All the other steps of the processes were the same. The product produced by the process of this invention was crisp and free-flowing. Products produced by the two processes described immediately above were noticeably less crisp and less free-flowing. When each of the above three products were stored in containers, pools of liquid were seen forming in the containers that contained the products produced by the separate spray-on of the liquid silicate and nonionic detergent. No such pools of liquid were noticed in the container containing the product of the process of this invention.

**EXAMPLE II**

When Example I was repeated with the exception of using 41 parts by weight of sodium silicate having a SiO$_2$:Na$_2$O ratio of 2.58:1 silicate in place of the sodium silicate mixture, substantially the same results were obtained.

**EXAMPLE III**

An agglomerated automatic dishwasher machine detergent composition having the following formulation was produced by the process of this invention as described in

<table>
<thead>
<tr>
<th>Example I</th>
</tr>
</thead>
</table>
| Sodium citrate dihydrate           | 28%  
| Sodium carbonate                   | 12%  
| Pluradot HA-433                    | 8%   
| Sodium silicate (SiO$_2$:Na$_2$O = 2.58:1) | 17%  
| Sodium silicate (SiO$_2$:Na$_2$O = 3.20) | 17%  
| Sodium sulfate                      | 18%  
|                                    | 100% |

The above product was crisp and free-flowing despite containing a high level of nonionic detergent.

When the process of this invention described in Example I is repeated using other nonionic detergents in place of the Pluradot HA-433 substantially the same results are obtained.

The addition of 3.5 parts by weight of potassium dichlorocyanurate to the agglomerates of Example I results in a product that cleans satisfactorily and has excellent spotting and filming characteristics. Similar performance results are obtained when 20 parts by weight of chlorinated trisodium polyphosphate is included with the sodium citrate and carbonate prior to spray-on of the liquid mixture.

What is claimed is:

1. A process for producing a crisp, free-flowing agglomerated granular detergent composition comprising the steps of:
   a. charging to a mixing zone particulate matter comprising an alkaline builder salt;
   b. agitating the particulate matter in the zone; and
   c. with continuous agitation spraying onto the particulate matter a liquid mixture having a temperature of from 110°F. to 200°F. of an alkali metal silicate having a SiO$_2$:alkali metal oxide ratio of from 3.2:1 to 2.5:1 and a nonionic detergent wherein the ratio of liquid alkali metal silicate to nonionic detergent ranges from 3:1 to 6:1 and wherein the liquid mixture is sprayed on at a level of from 25% to 50% based on the total composition to form said agglomerated granules.

2. The process of claim 1 wherein the nonionic detergent is sprayed on at a level sufficient to give the product a level of nonionic detergent of from 5% to 10%.

3. The process of claim 1 further comprising the step of adding a chlorine-yielding bleach to the composition in an amount sufficient to give the detergent composition an available chlorine content of from 0.1% to 10%.

4. The process of claim 3 wherein the chlorine-yielding bleach is a water-soluble salt of a dichlorocyanuric acid.

5. The process of claim 4 wherein the chlorine-yielding bleach is a water-soluble salt of a dichlorocyanuric acid.

6. The process of claim 1 wherein the alkaline builder salt is an alkali metal salt of citric acid.

7. The process of claim 6 wherein the nonionic detergent is a polyoxyalkylene nonionic detergent.

8. The process of claim 7 wherein the liquid alkali metal silicate contains from 45% to 70% water.

9. The process of claim 8 wherein the liquid mixture is continuously agitated prior to being sprayed onto the particulate matter.

10. The process of claim 9 wherein the liquid mixture is maintained at a temperature of from 140°F. to 160°F.