**EUROPEAN PATENT SPECIFICATION**

**EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention of the grant of the patent: 26.05.1999 Bulletin 1999/21

(21) Application number: 96905625.8

(22) Date of filing: 14.03.1996

(51) Int Cl. 6: C11D 17/00, C11D 3/06, C11D 7/06

(86) International application number: PCT/CA96/00153


(54) **PROCESS FOR FORMING TABLETED HIGH-CAUSTIC DETERGENT**

**VERFAHREN ZUR HERSTELLUNG VON HOCHALKALISCHEN WASCHMITTELTABLETTEN**

**PROCEDE DE FORMATION DE DETERGENTS EN COMPRIMES FORTEMENT CAUSTIQUES**

<table>
<thead>
<tr>
<th>Designated Contracting States:</th>
<th>CH DE ES FR GB IT LI NL SE</th>
</tr>
</thead>
</table>

| Priority: 22.03.1995 US 408538 |

| Date of publication of application: 07.01.1998 Bulletin 1998/02 |

| Proprietors: |
| UNILEVER N.V. 3013 AL Rotterdam (NL) Designated Contracting States: CH DE ES FR IT LI NL SE |
| UNILEVER PLC London EC4P 4BQ (GB) Designated Contracting States: GB |

| Inventors: |
| ROACH, Kenneth, James Canton, MI 48187 (US) |
| ANDERSON, Patricia Northville, MI 48167 (US) |

| Representative: |
| Rosen Jacobson, Frans Lucas M. et al Unilever N.V., Patent Division, P.O. Box 137 3130 AC Vlaardingen (NL) |

| References cited: |
| DE-A- 3 326 459 |

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).
Description

Background of the Invention

[0001] The institutional detergent market distributes a variety of products for washing silverware, pots and pans, dishes, floors, walls, stainless steel surfaces, tile and other areas.

[0002] Unlike products used in the home, institutional detergents are often sold in bulk and dispensed from mechanical dispensers. There are a variety of different physical forms these can take, including liquids, powders, solidified bricks, granules and tablets. Several factors enter into the determination of which particular physical form is most suitable for the desired application.

[0003] Feed rate is a very important consideration. With a liquid, where the product is directly injected for use, use concentration is easy to control. Unfortunately with liquids, the concentration of active components in the product is generally relatively low and therefore the container size can be prohibitively large. DE 3326459 discloses a liquid detergent for dishwashing which contains bicarbonate, metasilicates, phosphate, chloride, tensides, an acoustic source and water. EP 0297273 discloses a solid alkali cleaning material which contains alkali phosphate, silicates, carbonates and phosphoric partial ester. With solid forms, which are dissolved with water, the rate of dissolution will influence dispensing rate.

[0004] Delivering consistent feedstock is very important. With a brick formulation, the product consistency can be maintained to a certain extent, but dissolution rate can be slow and, as with many forms, there may also be problems with disposing of the container.

[0005] Another very important factor in distributing institutional detergents is packaging. For environment reasons, it is preferable to minimize packaging. U.S. Patent 5,076,301 discloses a bag of detergent tablets wherein the bag is a water soluble material. This product is apparently designed to minimize packaging, but has several significant disadvantages. Primarily, with a water soluble bag, the water will act to dissolve the plastic bag. However, the undissolved residue of such bags tend to clog the dispenser. Also with a water soluble bag, there is the requirement of an exterior overwrap to prevent humidity or extraneous water from destroying the water soluble bag during shipping and storage.

[0006] All of these problems are compounded with highly hygroscopic (highly caustic) and/or hydratable materials. Of course, with the caustic materials, the operators should never physically handle the detergent. Powdered cleaning compounds are typically dispersed with water. Given that premature exposure to water tends to increase the caking tendency of powders, clogging of the dispenser and uniform dispensing from powder systems, especially those prone to prolonged periods of inactivity, may be a problem.

[0007] Many detergents, particularly highly caustic detergents, dissolve in water and liberate a great deal of heat. It is therefore preferable to control the dissolution rate of these detergents to avoid temperature peaks in the dispensing equipment.

[0008] With tableted, high-caustic detergent, a further problem can be encountered. Anhydrous sodium hydroxide and potassium hydroxide are, of course, very hygroscopic. Typical detergent formulations generally include some free water, and certainly water of hydration from sources such as sodium tripolyphosphate hexahydrate. When tableting, the caustic comes into very close physical proximity to the water. The water is necessary for the tableting to occur at reasonable pressures. But once combined together, the caustic will exothermically react with the free water. For tableted high caustic detergents, if this reaction occurs after compression, the mechanical strength of the tablet will be reduced.

Summary of the Invention

[0009] Accordingly, it is an object of the present invention to provide a method of forming a tableted detergent which includes phosphate sequestrants, free water, and high levels of caustic. Further, it is an object of the present invention to provide such a product wherein the formed tablets do not deteriorate quickly after formation.

[0010] These objects and advantages of the present invention can be achieved by combining the individual components of the detergent, including the phosphate along with free water and caustic, in such a manner and/or order of addition that the overall temperature of the product at no time exceeds 75° C and preferably never exceeds 50° C and most preferably never exceeds 40° C. Careful blending, selection of raw materials and proper order of addition which factors in the hygroscopic nature of the materials and the liability of water, once absorbed, combine to achieve this result.

[0011] Accordingly, in one aspect the present invention provides a method of forming a tableted detergent from a formulation comprising a partially hydrated phosphate mixture including at least 10% hydrated phosphate based on the formulation, anhydrous caustic, 0.5% to 5% free water and 0 to 40% filler, said formulation including from 2 to 10% liquid components and from 2 to 10% by weight of water of hydration, the method comprising:

- adding said free water to said phosphate mixture without adding said filler, and allowing said water to be absorbed by said phosphate mixture; and
EP 0 815 195 B1

- subsequently adding 20 to 70% caustic to said phosphate mixture to form a second mixture whereby the temperature of said second mixture is maintained at less than 75°C, preferably less than 50 °C;

- compressing said second mixture to form tablets.

[0012] In this method according to the invention, it is preferred to add at least 5% filler to said phosphate mixture after said caustic is added to said phosphate mixture.

[0013] In a second aspect, the present invention provides a method of forming a compressed detergent tablet, said tablet comprising:

- from 20 to 70% by weight anhydrous caustic;

- from 20 to 60% by weight of a sequestering agent consisting of a combination of sodium tripolyphosphate and sodium tripolyphosphate hexahydrate said agent including at least 10% hydrated phosphate based on the formulation;

- from 0 to 4% by weight polycarboxylic acid having a molecular weight of 2,000 to 20,000;

- from 0 to 5% by weight of a defoaming agent, and from 0 to 4% by weight propylene glycol and from 5 to 40% filler;

- and free water, wherein said tablet includes from 2-10% liquid components and from 2 to 10% by weight of water hydration.

said method comprising combining said propylene glycol, said ethylene oxide, propylene oxide copolymer, said polycarboxylic acid and said free water to form a liquid mixture, combining said liquid mixture with said sequestering agents, and permitting said liquid mixture to be adsorbed by said sequestering agents to form a first detergent mixture;

- subsequently combining said filler and said caustic to said first detergent mixture to form a second detergent mixture and compacting said second detergent mixture to form tablets whereby the order of addition of the detergent components prevents the second detergent mixture from reaching a temperature in excess of 50 °C.

[0014] In a third aspect, the invention provides a method of forming a compressed tablet product comprising from 40 to 70% non-hydrated caustic; from 20 to 60% of a sequestering agent consisting of a mixture of sodium tripolyphosphate and sodium tripolyphosphate hexahydrate said mixture including at least 10% hydrated phosphate based on the formulation; from 5 to 20% filler, and from 1 to 5% free water, wherein said tablet product includes from 2 to 10% liquid components and from 2 to 10% by weight of water of hydration, said method comprising combining said water with said sequestering agent and allowing said water to be absorbed into said sequestering agent to form a detergent mixture, subsequently combining said caustic and said filler with said sequestering agent, thereby preventing the temperature of said detergent mixture from reaching 50 °C, compressing said detergent mixture to form tablets.

[0015] In one preferred embodiment of the present invention wherein fillers are included in the detergent formulation, the sequestering agents, i.e., sodium tripolyphosphate, anhydrous and hexahydrate, are combined together with any liquid components including all free water. After the liquid components are absorbed into the sequesterants, caustic, filler and any bleaching agent are added. The product can then be compressed to form tablets. In this manner, the hydration reaction is adequately controlled, i.e., the free water is absorbed by the species most capable of retaining it in the presence of caustic, thus reducing the potential for an exothermic reaction and subsequent deterioration of the tablet.

[0016] In an alternate embodiment of the present invention, cooling can be employed to physically control the temperature of the mixture, thereby preventing an undesirable excessively exothermic reaction. This, however, requires significant cooling time.

[0017] The objects and advantages of the present invention will be further appreciated in light of the following detailed description.

**Detailed Description**

[0018] The present invention is a method of making a high caustic tableted detergent, particularly a ware washing detergent. This ware washing detergent will include a source of caustic, a hardness sequestering system including a hydrated phosphate, low molecular weight water-soluble polymers, non-ionic defoaming surfactants, processing aids
and optionally bleaching sources.

[0019] The caustic source can be sodium or potassium hydroxide with sodium hydroxide preferred. Generally, for use in the present invention, this will include from about 20 to 70% caustic with about 45% to 57% caustic being preferred. The caustic is substantially anhydrous.

[0020] The hardness sequestering system can be a variety of different chemical components. The primary sequestants are alkali metal salts of polyphosphates. Optional sequestants include alkali metal salts of phosphonic acid and of gluconic acid, alkali metal salts of ethylene diamine tetraacetetic acid (EDTA), alkali metal salts of nitritriacetic acid (NTA) and alkali metal salts of polycarboxylic acids such as polyacrylic acid, polymaleic acid and mixtures thereof.

[0021] Phosphates are commonly available in anhydrous or hexahydrate forms. For purposes of the present invention, a mixture of anhydrous and hydrated phosphates is preferred. The composition should include at least 10% hydrated phosphate sequestant, based on total formulation.

[0022] Generally, the hardness sequestering system of the present invention will form 20 to 80% of the overall mass of the detergent composition, preferably 20 - 60% and more preferably 35 to 40%. A mixture of hydrated (hexahydrate) and anhydrous sodium tripolyphosphate in the mass ratio of 3:1 to 1:3 and preferably 1:1 to 2:1. In areas where the amount of phosphates is regulated, it may be necessary to supplement the water hardness control ability of the product by adding other sequestants such as the alkali metal salts of NTA or EDTA.

[0023] The present invention can optionally include a chlorine source. One preferred chlorine source is dichloroisocyanurate. This is added in amounts of up to 7% by weight. Other bleaching aids including alkali metal perborates and percarbonates may also be used.

[0024] In addition to the above, the detergent composition may include defoaming surfactants. One typical class of anionic defoaming surfactants is the phosphate esters. The defoaming non-ionic surfactant used herein is selected from the group consisting of alcohol alkoxylates, alkyl alkoxylates, block copolymers and mixtures thereof. Generally, these nonionic surfactants are prepared by the condensation reaction of a suitable amount of ethylene oxide and/or propylene oxide with a selected organic hydrophobic base under suitable oxyalkylation conditions. These reactions are well known and documented in the prior art. Generally, these will have a molecular weight of 900 to about 4,000. One such surfactant is an ethylene oxide propylene oxide block copolymer. Commercially available surfactants include Triton CF32*, Triton DF12*, Plurafac LF131*, Plurafac LF132*, Plurafac LF231*, Industrol N3* and Genapol PN30*. These can be included in an amount of about 0.5 to about 5% with about 1.5% preferred.

[0025] In addition to this, low molecular weight (2,000-20,000), water-soluble polybasic acids such as polyacrylic acid, polymaleic or polymethacrylic acid or copolymeric acids can be used as sequestering aids, to inhibit growth of calcium carbonate crystals and to improve rinseability. Preferably the water-soluble polymer will be a polycarboxylic acid such as polyacrylic acid having a molecular weight of around 5000. Generally, the present invention should include from about 1% to about 4% polyacrylic acid on an active basis with about 2% preferred.

[0026] The detergent formulation may also include 1% to 5% of a polyhydric water soluble alcohol. Suitable water soluble polyhydric alcohols include propylene glycol, ethylene glycol, polyethylene glycol, glycerine, pentaerythritol, trimethylol propane, triethanolamine, tri-isopropanol amine and the like. Propylene glycol is preferred, to be used in an amount of up to 4% by weight of the formulation. This acts as both a processing aid and a dissolution aid for the tablet, as is discussed below.

[0027] In order to provide a strong tablet the present invention will include from 2 to 10% liquid components, preferably less than 8%. Generally, this can be provided for by the nonionic surfactant, the polyalcohols and/or, free water. The formulation should also include 2% to 10% by weight of water of hydration. This also provides for a stronger tablet. Generally, there will be at least 0.5% up to 5% free water in the composition. This can be the solvent for the polymer or surfactant. It is preferable to keep the free water less than 5% and the total liquid at less than 10% to keep the product flowable and non-tacky during the tableting. When in the method of the invention the temperature of the second mixture is maintained at less than 50 °C, it is preferred to keep the free water content at 1 - 3%.

[0028] In addition to the above, the detergent formulation can include optional ingredients commonly referred to as fillers such as soda ash, the silicates such as sodium and potassium silicate and polysilicate, and sodium metasilicate and hydrates thereof, alkali metal chloride, alkali metal sulfates and alkali metal bicarbonate. These can be present in an amount of 1% to 30% by weight.

[0029] A preferred formulation for use in the present invention includes the following:

<table>
<thead>
<tr>
<th>Solid Components:</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.0%</td>
</tr>
<tr>
<td>21.0%</td>
</tr>
</tbody>
</table>

* Trade Marks
### Table 1 (continued)

<table>
<thead>
<tr>
<th>Solid Components:</th>
</tr>
</thead>
<tbody>
<tr>
<td>16.3% sodium tripolyphosphate powder</td>
</tr>
<tr>
<td>0.2% sodium dichloro-isocyanurate (ACL-60)</td>
</tr>
<tr>
<td>45.0% caustic bead</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Liquid Components:</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.5% 5000 molecular weight polyacrylic acid (48% active in water)</td>
</tr>
<tr>
<td>1.5% ethylene oxide propylene oxide block copolymer non-ionic surfactant</td>
</tr>
<tr>
<td>1.5% propylene glycol</td>
</tr>
</tbody>
</table>

[0030] In this formulation, the sodium tripolyphosphate hexahydrate provides 3.8% water of hydration and the polyacrylic acid provides about 2.3% free water.

[0031] A very high caustic formula includes:

### Table 2

<table>
<thead>
<tr>
<th>Solid Components:</th>
</tr>
</thead>
<tbody>
<tr>
<td>21.0% sodium tripolyphosphate hexahydrate (18% water of hydration)</td>
</tr>
<tr>
<td>16.3% sodium tripolyphosphate powder</td>
</tr>
<tr>
<td>56.7% caustic bead</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Liquid Components:</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.0% 5000 molecular weight polyacrylic acid (48% active in water)</td>
</tr>
<tr>
<td>1.5% ethylene oxide propylene oxide block copolymer non-ionic surfactant</td>
</tr>
<tr>
<td>1.5% propylene glycol</td>
</tr>
</tbody>
</table>

[0032] A third formulation which includes trisodium NTA is shown at Table 3.

### Table 3

<table>
<thead>
<tr>
<th>Solid Components:</th>
</tr>
</thead>
<tbody>
<tr>
<td>21.0% sodium tripolyphosphate hexahydrate (18% water of hydration)</td>
</tr>
<tr>
<td>16.3% anhydrous sodium tripolyphosphate</td>
</tr>
<tr>
<td>10.0% Trisodium NTA</td>
</tr>
<tr>
<td>1.7% soda ash</td>
</tr>
<tr>
<td>45.0% caustic</td>
</tr>
<tr>
<td>3.0% 5000 mw acrylic acid (48% active)</td>
</tr>
<tr>
<td>1.5% EOPO block copolymer</td>
</tr>
</tbody>
</table>

In order to formulate the detergent of the present invention, the phosphates are combined together and mixed in a ribbon or paddle blender. The fillers and other non-hygroscopic materials are not added at this time. Since a very low concentration of the liquid components is being added to the formulation, the liquid components should be combined prior to blending with the sequestants. Normally, the ethylene oxide propylene oxide block copolymer will react with the polycrylic acid to form a solid or gel. However, mixing the propylene glycol with these two liquid components prevents this reaction.

Thus, any liquid components such as polycrylic acid dissolved in water, the nonionic surfactant and the propylene glycol, are thoroughly mixed together and then sprayed evenly on the phosphate with mixing and allowed to soak into the phosphate. The caustic is added, then the fillers and finally the dichloroisocyanurate. If NTA or EDTA are added, these should generally be added with the fillers, i.e., after the caustic.

It is very important that during all stages of mixing, and even after formulation, the temperature be kept at less than 75°C, preferably less than about 50°C and preferably less than 40°C. It is theorized that hydration of the caustic generates heat which, if excessive, will cause the STPP hexahydrate to liberate water, most likely accompanied by the decomposition of the tripolyphosphate anion, which will generate more heat, weakening the tablet. However, by allowing the free water to be effectively completely absorbed by the phosphate, the hydration reaction is sufficiently slowed and excessive heat is not generated and the hexahydrate does not give up water. The phosphate mixture strongly bonds with the free water. As such when the caustic is combined with the mixture of phosphate and now bound water, hydration of the caustic is avoided. This prevention of caustic being hydrated in turn keeps the temperature down. Hence applicant's process provides a method of temperature control so as to maintain mixing temperature below 75°C by controlling or preventing the autocatalytic reaction.

If the free water is instead added to the fillers, or even to a mixture of filler and sequestant, then that water which hydrates the filler is relatively easily accessed by the caustic and the resulting hydration is so rapid, generating so much heat, that the hydrated phosphate gives up water causing the formed tablets to crumble or weaken.

The detergent blend is then pressed to form tablets using a standard tableting machine. One such machine suitable for use in the present invention is the Stokes brand tableter. Generally, to form tablets, the powder is subjected to 4 to 10 tons pressure. Generally, the tablet will have a thickness of about 12 to 13 mm and a diameter of about 20 mm. The maximum diameter will be a function of the dispenser/feed water interface area.

The tablets of the end product after being produced do not weaken significantly over time. These can then be used in a typical ware washer apparatus equipped with a water spray detergent dispenser.

There are alternate methods to achieve the same result. An initial method of achieving this result is to omit fillers and form the detergent with anhydrous and hydrated sequestants, along with the previously mentioned liquid components, as shown in Table 2. The phosphates are combined with the liquid component so that any free water present is adsorbed onto the sequestants. The caustic is then added and the mixture tableted. Again, because the phosphates hold the water relatively tightly, the temperature at all times is maintained at less than 75°C and generally less than 50°C and therefore the hexahydrate will not liberate water which can react with the caustic. The formed tablets do not deteriorate rapidly after formation.

In a second alternate method of practicing the invention, the fillers can be combined with the phosphates and the water subsequently added. This can then be blended together with the caustic, provided sufficient cooling is provided so that the temperature is kept less than 50°C and preferably less than 40°C. This temperature is maintained for sufficient time to allow the caustic to react completely with any labile water prior to the tableting operation. Of course, this requires added processing time.

By employing the preferred method, the formed tablets have a drastically improved storage stability and shelf life. The end products are stronger, which means they are less likely during shipping to break apart and during use they will dissolve more slowly and evenly, providing for an even distribution of the detergent dissolved in water without creating an extreme exotherm. In all, this system provides many unique advantages and although several embodiments of the present invention have been disclosed, the invention itself should be defined only by the appended claims wherein we claim:

Claims

1. A method of forming a tableted detergent from a formulation comprising a partially hydrated phosphate mixture, including at least 10% hydrated phosphate based on the formulation, anhydrous caustic, 0.5% to 5% free water and 0 to 40% filler said formulation including from 2 to 10% liquid components and from 2 to 10% by weight of
water of hydration, the method comprising:
adding said free water to said phosphate mixture without adding said filler, and allowing said water to be absorbed by said phosphate mixture;
and subsequently adding 20% to 70% caustic to said phosphate mixture to form a second mixture whereby the temperature of said second mixture is maintained at less than 75° C;
compressing said second mixture to form tablets.

2. The method claimed in claim 1 wherein at least 5% filler is added to said phosphate mixture after said caustic is added to said phosphate mixture.

3. The method claimed in claim 1 wherein said temperature of said second mixture is maintained at less than 50° C.

4. The method claimed in claim 2 wherein said hydrated sequestrant comprises a mixture of sodium tripolyphosphate (STPP) and STPP hexahydrate.

5. The method claimed in claim 3 comprising adding fillers to said phosphate mixture after said water has been absorbed by said phosphate.

6. The method claimed in claim 3 comprising 1% to 3% free water.

7. The method claimed in claim 2 wherein said filler is selected from the group consisting of soda ash, alkali metal silicates, alkali metal polysilicates, alkali metal metasilicates, alkali metal chloride, alkali metal sulfates, and alkali metal bicarbonates.

8. The method claimed in claim 3 wherein the temperature of said second mixture is maintained at less than 40° C.

9. A method of forming a compressed detergent tablet, said tablet comprising:

from 20% to 70% by weight anhydrous caustic;
from 20% to 60% by weight of a sequestering agent consisting of a combination of sodium tripolyphosphate and sodium tripolyphosphate hexahydrate said agent including at least 10 % hydrated phosphate based on the formulation:
from 0 to 4% by weight polycarboxylic acid having a molecular weight of 2,000 to 20,000;
from 0 to 5% by weight of a defoaming agent, and from 0 to 4% by weight propylene glycol and from 5% to 40% filler;
and free water, wherein said tablet includes from 2-10 % liquid components and from 2 to 10 % by weight of water of hydration;
said method comprising combining said propylene glycol, said ethylene oxide, propylene oxide copolymer, said polycarboxylic acid and said free water to form a liquid mixture, combining said liquid mixture with said sequestrating agents, and permitting said liquid mixture to be adsorbed by said sequestering agents to form a first detergent mixture;
subsequently combining said filler and said caustic to said first detergent mixture to form a second detergent mixture and compacting said second detergent mixture to form tablets whereby the order of addition of the detergent components prevents the second detergent mixture from reaching a temperature in excess of 50°C.

10. A method of forming a compressed tablet product comprising from 40% to 70% non-hydrated caustic; from 20% to 60% of a sequestering agent consisting of a mixture of sodium tripolyphosphate and sodium tripolyphosphate hexahydrate said mixture including at least 10 % hydrated phosphate based on the formulation; from 5% to 20% filler, and from 1% to 5% free water, wherein said tablet product includes from 2 to 10 % liquid components and from 2 to 10 % by weight of water of hydration, said method comprising combining said water with said sequestering agent and allowing said water to be absorbed into said sequestering agent to form a detergent mixture, subsequently combining said caustic and said filler with said sequestering agent, thereby preventing the temperature of said detergent mixture from reaching 50° C, compressing said detergent mixture to form tablets.
Patentansprüche

1. Ein Verfahren zur Bildung eines tablettierten Waschmittels aus einer Formulierung, enthaltend eine partiell hydratisierte Phosphatmischung, enthaltend zumindest 10 % hydratisiertes Phosphat, basierend auf der Formulierung, wasserfreies Ätzmittel, 0,5 % bis 5 % nichtgebundenes Wasser und 0 bis 40 % Füllstoff, wobei die erwähnte Formulierung von 2 bis 10 % flüssige Komponenten und von 2 bis 10 Gewichtsprozent Hydratationswasser enthält, umfassend:

Zusatz des erwähnten nichtgebundenen Wassers zu der erwähnten Phosphatmischung ohne Zugabe des erwähnten Füllstoffs, und Ermöglichen dem erwähnten Wasser durch die erwähnte Phosphatmischung absorbiert zu sein;

und anschließend Zusatz von 20 % bis 70 % Ätzmittel zu der erwähnten Phosphatmischung zur Bildung einer zweiten Mischung, wobei die Temperatur der erwähnten zweiten Mischung auf weniger als 75°C gehalten ist;

Zusammenpressen der erwähnten zweiten Mischung zur Bildung von Tabletten.

2. Das Verfahren gemäß Anspruch 1, worin zumindest 5 % Füllstoff zu der erwähnten Phosphatmischung zugesetzt ist, nachdern das erwähnte Ätzmittel zu der erwähnten Phosphatmischung zugesetzt wurde.

3. Das Verfahren gemäß Anspruch 1, worin die erwähnte Temperatur der erwähnten zweiten Mischung auf weniger als 50°C gehalten wird.


5. Das Verfahren gemäß Anspruch 3, umfassend das Zusatz von Füllstoffen zu der erwähnten Phosphatmischung, nachdern das erwähnte Wasser durch das erwähnte Phosphat absorbiert worden ist.

6. Das Verfahren gemäß Anspruch 3, enthaltend 1 % bis 3 % nichtgebundenes Wasser.


8. Das Verfahren gemäß Anspruch 3, worin die Temperatur der erwähnten zweiten Mischung auf weniger als 40°C gehalten wird.

9. Ein Verfahren zur Bildung einer verdichteten Waschmitteltablette, wobei die Tablette enthält:

Von 20 % bis 70 Gewichtsprozent wasserfreies Ätzmittel;

von 20 % bis 60 Gewichtsprozent eines Sequestrierungsmittels, bestehend aus einer Kombination von Natriumtripolphosphat und Natriumtripolphosphat Hexahydrat, wobei das erwähnte Mittel zumindest 10 % hydratisiertes Phosphat auf Basis der Formulierung enthält;

von 0 bis 4 Gewichtsprozent Polycarbon säure mit einem Molekulargewicht von 2000 bis 20 000;

von 0 bis 5 Gewichtsprozent eines Antischäummittels, und von 0 bis 4 Gewichtsprozent Propylen glykol und von 5 % bis 40 % Füllstoff;

und nichtgebundenes Wasser, worin die erwähnte Tablette von 2 bis 10 % flüssige Komponenten und von 2 bis 10 Gewichtsprozent Hydratationswasser enthält; wobei das erwähnte Verfahren umfaßt das Kombinieren des erwähnten Propylen glykols, des erwähnten Ethylenoxids, des Propylenoxid-Copolymeren, der erwähnten Polycarbon säure und des erwähnten nichtgebundenen Wassers zur Bildung einer flüssigen Mischung, das Kombinieren der erwähnten flüssigen Mischung mit den erwähnten Sequestrierungsmitteln, und es der erwähnten flüssigen Mischung ermöglicht, durch die erwähnten Sequestrierungsmittel adsorbiert zu sein zur Bildung einer ersten Waschmittelmischung; anschließend Kombinieren des erwähnten Füllstoffs und des erwähnten Ätzmittels zu der erwähnten ersten Waschmittelmischung zur Bildung einer zweiten Waschmittelmischung und Zusammenrollen der erwähnten zweiten Waschmittelmischung zur Bildung von Tabletten, wobei die Reihenfolge der Zugabe der Waschmittelkomponenten die zweite Waschmittelmischung vom Erreichen einer Temperatur von mehr als 50°C abhält.
10. Ein Verfahren zur Bildung eines verdichteten Tablettenprodukts, enthaltend von 40 % bis 70 % nicht-hydratisiertes Ätzmittel; von 20 % bis 60 % eines Sequestrierungsmittels, bestehend aus einer Mischung von Natriumtripolyphosphat und Natriumtripolyphosphat-Hexahydrat, wobei die erwähnte Mischung zumindest 10 % hydratisiertes Phosphat, basierend auf der Formulierung, enthält; von 5 % bis 20 % Füllstoff, und von 1 % bis 5 % nichtgebundenenes Wasser, worin das erwähnte Tablettenprodukt von 2 bis 10 % flüssige Komponenten und von 2 bis 10 Gewichtsprozent Hydrationswasser enthält, wobei das erwähnte Verfahren das Kombinieren des erwähnten Wassers mit dem erwähnten Sequestrierungsmittel umfasst, und es dem erwähnten Wasser erlaubt, in das erwähnte Sequestrierungsmittel absorbiert zu werden zur Bildung einer Waschmittelwirkung, anschließend Kombinieren des erwähnten Ätzmittels und des erwähnten Füllstoffes mit dem erwähnten Sequestrierungsmittel, dadurch verhindert es das Erreichen der erwähnten Waschmittelwirkung auf eine Temperatur von 50°C, Verdichten der erwähnten Waschmittelwirkung zur Bildung von Tabletten.

Revendications

1. Un procédé pour la formation d'un détergent, conditionné en comprimés, à partir d'une formulation comportant un mélange de type phosphate partiellement hydraté, comportant au moins un phosphate hydraté à 10 %, en se basant sur la formulation, un produit caustique anhydre, de 0,5 % à 5 % d'eau non combinée et de 0 à 40 % de charge, cette formulation comportant de 2 à 10 % de composants liquides et de 2 à 10 % en poids d'eau d'hydratation, le procédé comportant :

- l'addition de ladite eau non combinée audit mélange de type phosphate sans ajouter ladite charge, après quoi on laisse ladite eau être absorbée par ledit mélange de type phosphate ; et
- l'addition subéquente de 20 à 70 % de produit caustique audit mélange de phosphate afin de former un second mélange de sorte que la température dudit second mélange est maintenue à moins de 75°C ;
- la compression dudit second mélange afin de former des comprimés.

2. Le procédé revendiqué dans la revendication 1, dans lequel au moins 5 % de charge sont ajoutés au mélange de type phosphate après que ledit produit caustique ait été ajouté audit mélange de type phosphate.

3. Le procédé revendiqué dans la revendication 1, dans lequel ladite température dudit second mélange est maintenue inférieure à 50°C.

4. Le procédé revendiqué dans la revendication 2, dans lequel ledit agent sequestrant hydraté comporte un mélange de tripolyphosphate de sodium (STPP) et de STPP hexahydraté.

5. Le procédé revendiqué dans la revendication 3, comportant l'addition de charges audit mélange de type phosphate une fois que ladite eau a été absorbée par ledit phosphate.

6. Le procédé revendiqué dans la revendication 3, comportant de 1 % à 3 % d'eau non combinée.

7. Le procédé revendiqué dans la revendication 2, dans lequel ladite charge est choisie dans le groupe constitué du carbonate de sodium anhydre, des silicates de métaux alcalins, des polysilicates de métaux alcalins, des métasilicates de métaux alcalins, des chlorures de métaux alcalins, des sulfates de métaux alcalins, et des bicarbonates de métaux alcalins.

8. Le procédé revendiqué dans la revendication 3, dans lequel la température dudit second mélange est maintenue inférieure à 40°C.

9. Un procédé pour former un comprimé détergent, ledit comprimé comportant :

- de 20 à 70 % en poids de produit caustique anhydre ;
- de 20 à 60 % en poids d'un agent sequestrant, constitué d'une combinaison de tripolyphosphate de sodium et de tripolyphosphate de sodium hexahydraté, ledit agent comportant au moins 10 % de phosphate hydraté, en se basant sur la formulation ;
- de 0 à 4 % en poids d'acide polycarboxylique, ayant un poids moléculaire de 2 000 à 20 000 ;
- de 0 à 5 % en poids d'un agent antimousse et de 0 à 4 % en poids de propylène glycol et de 5 à 40 % de charge ;
et de l'eau non combinée, ledit comprimé renfermant de 2 à 10 % de composants liquides et de 2 à 10 % en poids d'eau d'hydratation, ledit procédé consistant à combiner ledit propylène glycol, ledit oxyde d'éthylène, ledit copolymère d'oxyde de propylène, ledit acide polycarboxylique et ladite eau non combinée afin de réaliser un mélange liquide, de combiner ledit mélange liquide avec des agents sequestrants, tout en laissant ledit mélange liquide être absorbé par lesdits agents sequestrants afin de former un premier mélange détergent ;
de combiner par la suite ladite charge et ledit produit caustique audit premier mélange détergent afin de réaliser un second mélange détergent et de compacter ledit second mélange détergent pour former des comprimés, de sorte que l'ordre d'addition des composants détergents évite que le second mélange détergent n'atteigne une température supérieure à 50°C.

10. Un procédé pour former un produit de type comprimé, comportant de 40 à 70 % d'un produit caustique non-hydraté, de 20 à 60 % d'un agent sequestrant, constitué d'un mélange de tripolyphosphate de sodium et de tripolyphosphate de sodium hexahydraté, ledit mélange comportant au moins 10 % de phosphate hydraté, en se basant sur la formulation : de 5 à 20 % de charge, et de 1 à 5 % d'eau non combinée, ledit produit de type comprimé comportant de 2 à 10 % de composants liquides et de 2 à 10 % en poids d'eau d'hydratation, ce procédé consistant à combiner ladite eau avec ledit agent sequestrant tout en laissant l'eau être absorbée dans ledit agent sequestrant pour former un mélange détergent, en combinant par la suite ledit produit caustique et ladite charge avec ledit agent sequestrant, de façon à éviter que la température dudit mélange détergent n'atteigne 50°C, en comprimant ledit mélange détergent pour former des comprimés.