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Process for the manufacture of spray-dried detergent powder.

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The present invention relates to a process for the production of detergent powders containing anionic detergent-active compounds, by slurry-making and spray-drying.

Spray-dried detergent powders are generally produced by preparing an aqueous slurry of detergent-active compounds, builder and other salts, sodium silicate, fluorescers and other non-heat-sensitive ingredients, and then spray-drying the slurry to form a free-flowing powder. Ingredients unsuitable for spray-drying, such as bleaches, perfumes and enzymes, may be postdosed subsequently to the spray-dried base powder.

Anionic detergent-active compounds such as alkylbenzene sulphonates are incorporated in spray-dried detergent powders via the slurry. Alkylbenzene sulphonates are manufactured by sulphonation of the corresponding alkylbenzene to give a sulphonic acid which can then be neutralised with a suitable base, for example, sodium or potassium hydroxide, sodium carbonate, ammonia or triethanolamine, to give the salt of the desired cation. The sodium salt, which is the most commonly used salt in detergent powders, is generally prepared by neutralisation with aqueous sodium hydroxide solution to yield a paste containing 50% or more water, and that material can be incorporated directly in the slurry.

It has already been proposed to omit a separate neutralisation step by dosing the alkylbenzene sulphonic acid directly into the slurry-making vessel and neutralising it there with sodium hydroxide solution: this procedure is disclosed, for example, in GB-A-151767 (Colgate-Palmolive) and GB-A-1355187 (Unilever). The heat liberated in the neutralisation reaction is useful for heating the slurry to the desired temperature which is generally about 80°C. The alkylbenzene sulphonic acid is conventionally one of the first ingredients introduced into the slurry-making vessel, and is neutralised with sodium hydroxide solution before other ingredients are added. It has hitherto been believed that it is essential to neutralise the sulphonic acid before any sodium silicate is added, in order to prevent those components from interacting to form relatively insoluble siliceous compounds which could seriously affect the solubility of the finished detergent powder and could also lead to jet blocking during the spray-drying operation.

It has now been found that it is possible to delay the addition of sulphonic acid until a later stage in the slurry-making process without encountering this disadvantage. The new procedure gives both product and process benefits, especially in the manufacture of powders containing a relatively high level of anionic detergent-active material.

DEFINITION OF THE INVENTION

The present invention provides a process for the manufacture of a particulate detergent composition, which comprises the steps of:
(i) preparing an aqueous slurry comprising one or more anionic non-soap detergent-active compounds, and one or more alkaline inorganic salts, and
(ii) spray-drying the slurry to form a powder;

wherein in step (i) an anionic detergent-active compound is incorporated in wholly or predominantly acid form into the slurry after the incorporation of alkaline material in an amount such that a total of at least two equivalents of alkaline material per equivalent of the said acidic detergent-active compound are present, with the proviso, that sodium carbonate is not used as the only alkaline inorganic salt.

DESCRIPTION OF THE INVENTION

A significant characteristic of the invention is the incorporation of an anionic detergent-active compound wholly or predominantly in its acid form at a relatively late stage in the slurry-making process, when a large reservoir of alkaline material is already present in the slurry.

The invention is applicable to the incorporation of any anionic detergent-active compound having a stable acid form. It is of especial interest in relation to sulphonic acids, especially alkylaryl sulphonic acids,
more especially C₈-C₁₅ alkylbenzene sulphonates, both linear and branched. It may be advantageous for the sulphonate to be partially neutralised, for example, up to 10% by weight neutralised, before introduction into the slurry-making vessel: a neutralisation level of about 5% appears to be especially beneficial. This can be helpful in stabilising the sulphonate and preventing it from darkening in colour during storage. When the sodium salt of the sulphonate is desired, such preneutralisation is conveniently carried out with aqueous sodium hydroxide solution. For convenience, acid or partially neutralised acid added to the slurry will be referred to hereinafter simply as "sulphonic acid".

At the time at which the sulphonate acid is introduced into the slurry-making vessel, there must already be present at least twice as much alkaline material as is required fully to neutralise the sulphonate acid. This will generally be constituted in part by a base such as sodium hydroxide which is especially intended for the purpose, in part by alkaline sodium salts present as builders, and possibly in part by alkaline sodium silicate. It is normally desirable to include in the slurry sufficient sodium hydroxide solution or other alkali to neutralise the sulphonate acid fully but in the manufacture of low-pH detergent powders intended for washing delicate fabrics it may be preferable to use a less than stoichiometric quantity of alkali and allow the neutralisation reaction to be completed by the alkaline builder salts present.

It is even possible for neutralising alkali to be omitted completely and the neutralisation reaction to be effected entirely by means of alkaline salts.

If the detergent composition prepared in accordance with the invention is to include alkaline sodium silicate, the silicate is preferably incorporated in the slurry before the sulphonate acid, and thus contributes to the alkaline material, mentioned previously, that must be present in at least a twofold amount relative to the sulphonate acid at the time when the latter component is added.

Preferred examples of alkaline builder salts present in the slurry when the sulphonate acid is added are sodium tripolyphosphate, sodium carbonate and mixtures of these. These materials are commonly used in detergent powders as builders which sequester or precipitate calcium and magnesium salts in the wash liquor. They generally comprise at least 25% by weight of the detergent powder so that if they are incorporated in the slurry before addition of the sulphonate acid they can serve as a large alkaline buffer to prevent or minimise any adverse interaction between sodium silicate and sulphonate acid. Efficient stirring in the slurry-making vessel also helps to eliminate such adverse interaction.

Any alkali added to the slurry specifically for neutralisation of the sulphonate acid of course makes an additional contribution to the reservoir of alkaline material in the slurry. In the case of alkylbenzenesulphonic acid, the amount of sodium hydroxide required to neutralise the sulphonate acid completely generally ranges from 10 to 20g per 100g sulphonate acid. This relatively large variation occurs because the amount of sulphuric acid present as a by-product in the sulphonate acid can vary quite widely: it is likely to be higher in sulphonate acids prepared by oleum sulphonation than in sulphonate acids prepared by sulphur trioxide sulphonation. Sodium hydroxide solution of specific gravity 1.5 is conveniently used as the neutralising alkali.

When a neutralising alkali is used, it need not be introduced into the slurry before the sulphonate acid, provided that sufficient alkaline material from other sources is present when the sulphonate acid is added. It may be preferable to add the alkali and sulphonate acid simultaneously.

The process of the invention gives several worthwhile advantages. One has already been mentioned: if sodium silicate is present, interaction between sodium silicate and sulphonate acid to form insoluble species can be minimised. Sodium silicate is a desirable ingredient in detergent compositions because it improves powder structure, prevents corrosion of washing machine parts in use, and provides building capacity (towards magnesium ions). The process of the invention is, however, also beneficial for the production of silicate-free powders and gives other advantages applicable both to silicate-containing and to silicate-free powders.

Better control of the slurry temperature during the in-situ sulphonation step can be achieved than in the prior art process when the sulphonate acid is neutralised before addition of the builder salts and other components, because of the larger mass of material present, and available to act as a heat sink, at the time of the addition of the sulphonate acid. This benefit is especially noticeable when spray-dried powders containing 20% by weight or more of alkylaryl sulphonate are prepared: using the prior art procedure, so much heat may be generated under those circumstances that the slurry can boil over.

A less aerated slurry is also obtained by the process of the invention, especially when other, high-foaming anionic, surfactants such as alkyl sulphates or alkyl ether sulphates are absent. A less aerated slurry gives a spray-dried powder of higher particle density, and allows high bulk density powders to be prepared without the need for a separate deaeration step. A less aerated slurry is also less bulky so that larger batches of slurry can be processed in a given slurry-making vessel: for powders containing high levels of alkylbenzene sulphonate (20% or more) an increase in batch size of up to 20% has been

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achieved, leading to a corresponding increase in the output of the slurry making plant. Pumping of a less aerated slurry is also easier because its viscosity is lower and cavitation problems are less likely to occur.

Another benefit of the process of the invention is seen when sodium tripolyphosphate is a major building and structuring component of the powder: in a preferred embodiment of the invention, all sodium tripolyphosphate is added before the addition of the sulphonic acid. The temperature of the slurry at the time at which the sodium tripolyphosphate is added is then lower than in the prior art process in which the sulphonic acid neutralisation step has already occurred and raised the slurry temperature. The sodium tripolyphosphate can dissolve and hydrate more quickly at a lower temperature: this ensures that less degradation of the tripolyphosphate occurs during spray-drying, and also favours the growth of very small sodium tripolyphosphate hexahydrate crystals in the slurry, thus giving spray-dried powders of improved structure and absorptivity. Advantageously, a period of at least 2 minutes may be allowed to lapse between addition of the sodium tripolyphosphate and addition of the sulphonic acid to the slurry, in order to maximise hydration of the sodium tripolyphosphate before any temperature increase due to the neutralisation reaction occurs.

In a typical slurry-making plant, the finished slurry is pumped, first via low-pressure pipes and then via high-pressure pipes, to a distribution manifold for the atomising nozzles of a spray-drying tower. As an alternative to incorporating the sulphonic acid in the slurry-making vessel itself, it may be injected either into the low-pressure pump or pipes, or into the high-pressure pump or pipes. It has been found especially beneficial to inject the sulphonic acid into the high-pressure pump or pipes, very shortly before the slurry arrives at the spray-nozzle distribution manifold.

In this procedure slurry aeration and interaction with other materials in the slurry, particularly other detergent-active materials, are minimised: this is of especial advantage if nonionic surfactants are present, since alkylbenzene sulphonates can form viscous phases with nonionic surfactants in slurries, especially when the weight ratio of alkylbenzene sulphonate to nonionic surfactant is within the range of from 1:5 to 5:1. Furthermore, the slurry viscosity is lower before the addition of the sulphonic acid, and this lower viscosity can be exploited to the full, in easier pumping operations, if the sulphonic acid addition is delayed as long as possible.

Alkylbenzene sulphonics are fairly viscous liquids at ambient temperatures but can be pumped without too much difficulty using positive displacement pumps. It has been found that sulphonic acid can be used at ambient temperature if it is to be added to the slurry-making vessel. When the alternative procedure of injection into a slurry transfer pump or pipe is to be used, however, the sulphonic acid is preferably heated to a temperature of about 50 to 60°C to lower its viscosity to a value at which accurate metering with a pump of the piston type is possible. An alternative method of reducing the viscosity of the sulphonic acid is to premix it with a viscosity-lowering ingredient, especially a nonionic surfactant, but this method is suitable only for preparing powders in which the viscosity-lowering ingredient is required.

The process of the invention may be used with advantage to prepare any spray-dried detergent powder containing an anionic detergent-active compound having a stable acid form. This will normally be a sulphonate-type anionic detergent, especially an alkylaryl sulphonate and more especially a C_{12}-C_{15} linear alkylbenzene sulphonate. Other anionic detergent-active compounds may also be present, for example, primary and secondary alkyl sulfates, olefin sulphonates, alkane sulphonates, alklyl ether sulphates, and fatty acid ester sulphonates.

Nonionic surfactants may also be used in detergent powders prepared according to the invention. These include the primary and secondary alcohol ethoxylates, especially the C_{12}-C_{15} primary and secondary alcohols ethoxylated with an average of from 3 to 20 moles of ethylene oxide per mole of alcohol.

It may also be desirable to include one or more soaps of fatty acids. The soaps which can be used are preferably sodium soap derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil.

The total amount of detergent-active material (surfactant), excluding soap, in detergent powders prepared according to the invention is generally within the range of from 5 to 40% by weight. For low-sudsing powders intended for use in European front-loading automatic washing machines the preferred range is from 5 to 20% by weight, with a weight ratio of anionic surfactant to nonionic surfactant not exceeding 10:1, and preferably not exceeding 8:1. As indicated above, however, the present invention is especially applicable to the manufacture of medium- and high-sudsing powders containing higher levels of anionic surfactant and/or higher ratios of anionic surfactant to nonionic surfactant.

Detergent powders prepared in accordance with the invention will also comprise one or more detergency builders, suitably in an amount of from 10 to 60% by weight. Detergency builders are very well known to those skilled in the art and include sodium tripolyphosphate, orthophosphate and pyrophosphate;
crystalline and amorphous sodium aluminosilicate; sodium carbonate; and monomeric and polymeric polycarboxylates, for example, sodium citrate, nitritotriacetate and polyacrylate, and acrylic copolymers. As mentioned above, at least some of the builders incorporated in the powder must be alkaline salts.

Other inorganic salts without a detergency building function, for example, sodium sulphate, may also be included in detergent powders prepared according to the invention.

Detergent powders prepared according to the invention will also generally contain various additives to enhance the efficiency of the product, notably bleach systems, antiredeposition agents, fluorescers, lather suppressors, enzymes and perfumes. These may be added to the slurry or postdosed into the spray-dried powder in accordance with their known suitability for undergoing spray-drying processes.

EXAMPLES

The invention will now be illustrated in further detail by the following non-limiting Examples.

Example 1

A slurry was prepared in a batch slurry-making apparatus from the following ingredients, added in the order listed:

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Parts by Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>1814</td>
</tr>
<tr>
<td>Sodium silicate solution (1.6 S.G.)</td>
<td>529</td>
</tr>
<tr>
<td>EDTA 40% solution</td>
<td>13</td>
</tr>
<tr>
<td>Fluorescer premix slurry</td>
<td>38</td>
</tr>
<tr>
<td>Sodium carboxymethyl cellulose</td>
<td>19</td>
</tr>
<tr>
<td>Sodium tripolyphosphate</td>
<td>1475</td>
</tr>
<tr>
<td>Sodium hydroxide solution (1.5 S.G.)</td>
<td>64</td>
</tr>
<tr>
<td>Sodium sulphate</td>
<td>540</td>
</tr>
<tr>
<td>Linear alkylbenzene sulphonic acid</td>
<td>241</td>
</tr>
<tr>
<td>(Petrelab (Trade Mark) 550 ex Petresa)</td>
<td>4733</td>
</tr>
</tbody>
</table>

The contents of the slurry-making vessel were stirred continuously during the preparation of the slurry. During addition of the sulphonic acid, which was at ambient temperature, the temperature of the slurry rose from about 80°C to about 85°C. It was noticeable that much less air than usual was entrained in the slurry during its preparation.

It will be noted that at the time of addition of the alkylbenzene sulphonic acid (746 equivalents) to the slurry, 16782 equivalents of alkaline material (16033 equivalents from the sodium tripolyphosphate and 749 equivalents from the sodium hydroxide) were available in the slurry-making vessel, that is to say, the equivalent ratio of alkali to sulphonic acid was 22:1 without counting the sodium silicate and minor ingredients.

The slurry was transferred via low and high-pressure transfer pipes to a spray-drying tower, 162 parts of a nonionic surfactant being injected into the high-pressure slurry transfer pipe just before it entered the spray jet distribution manifold.

The solubility of the powder was assessed by stirring 4g into 400 ml of water at a temperature of 40°C for 2 minutes and then filtering the undissolved material. The weight of undissolved material under these test conditions should be less than 0.16g for a detergent powder, equivalent to 4% undissolved powder; the weight of undissolved material is greatly affected by the amount of insoluble siliceous material present.

Under the above testing conditions, the level of undissolved material in the detergent powder of
Example 1 was very satisfactory at 1.1%. The sulphonic acid used in Example 1 had been prepared by sulphonating an alkylbenzene with sulphur trioxide and air on a falling film reactor. It was of very good quality and contained only 0.9% by weight of free sulphuric acid.

Example 2

This Example describes the preparation of a high-sudsing detergent powder containing 20% by weight of alkylbenzene sulphonate. The alkylbenzene sulphonamic acid used had been prepared by reacting the corresponding alkylbenzene directly with 20% oleum and then allowing the resulting mixture of sulphonamic and sulphuric acids to separate into two layers; its sulphuric acid content was 6.9% by weight.

A slurry was prepared in a batch slurry-making vessel by adding the following ingredients with mixing, in the order stated:

<table>
<thead>
<tr>
<th>Parts by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
</tr>
<tr>
<td>Sodium silicate solution (1.6 S.G.)</td>
</tr>
<tr>
<td>Flourescer premix</td>
</tr>
<tr>
<td>Sodium carboxymethyl cellulose</td>
</tr>
<tr>
<td>Sodium tripolyphosphate</td>
</tr>
<tr>
<td>Sodium hydroxide solution (1.5 S.G.)</td>
</tr>
<tr>
<td>Alkylbenzene sulphonamic acid</td>
</tr>
<tr>
<td>(as Example 1</td>
</tr>
<tr>
<td>Sodium sulphate</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

It will be noted that at the time of addition of the alkylbenzene sulphonamic acid (3525 equivalents) to the slurry, 18155 equivalents of alkaline material from the sodium tripolyphosphate and the sodium hydroxide were available, that is to say, the equivalent ratio of alkali to sulphonamic acid was 4.8:1.

The slurry temperature before addition of the sodium tripolyphosphate was 56°C and this rose to 72°C after a 2-minute period to allow the sodium tripolyphosphate to hydrate: this temperature rise indicated that 93% hydration has been achieved. During addition of the alkylbenzene sulphonamic acid the temperature rose by a further 22°C to a final value of 94°C; at no time during the slurry-making process was heat from an external source applied to the vessel.

The degree of aeration of the slurry after all the slurry ingredients had been added was significantly less than normal. The slurry was easy to pump.

Without deaeration the slurry was transferred to a spray-drying tower and spray-dried to give a free-flowing powder having a bulk density of 413 g/litre.

The standard dissolution test described in Example 1 established that the dissolving properties of the detergent powder were very good. The amount of undissolved material remaining after 2 minutes' stirring was only 0.1%. This result confirmed that no adverse reaction between the sulphonamic acid and the sodium silicate had occurred in the slurry.

Comparative Example A

The same ingredients as in Example 2, in the same proportions, were dosed into an identical slurry-making vessel in the following order:
## Example 3

This Example illustrates an alternative procedure according to the invention, in which alkylbenzene sulphonic acid was injected continuously into the slurry immediately before the slurry reached the distribution manifold for the spray nozzles of the spray-drying tower.

In a batch slurry-making vessel a slurry was prepared from the following ingredients, added with thorough mixing in the order listed:

<table>
<thead>
<tr>
<th>Parts by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
</tr>
<tr>
<td>Sodium silicate solution (1.6 S.G.)</td>
</tr>
<tr>
<td>EDTA solution (40%)</td>
</tr>
<tr>
<td>Sodium carboxymethyl cellulose</td>
</tr>
<tr>
<td>Fluorescer premix</td>
</tr>
<tr>
<td>Polyacrylate polymer solution (25%)</td>
</tr>
<tr>
<td>Nonionic surfactant</td>
</tr>
<tr>
<td>Sodium tripolyphosphate</td>
</tr>
<tr>
<td>Sodium hydroxide solution (1.5 S.G.)</td>
</tr>
<tr>
<td><strong>Total</strong></td>
</tr>
</tbody>
</table>

The slurry was pumped through low and high-pressure pipes to the distribution manifold. The alkylbenzene sulphonic acid used in Example 1 (689 parts) was injected continuously at a pressure of 50
bar into the slurry just before it entered the manifold: the slurry pressure at this point was 45 bar. A simple static in-line mixer installed after the injection point ensured that the sulphanic acid mixed well with the slurry. Further and more intense local mixing also took place in the high shear swirl type of spray jets in the spray-drying tower.

At the point of injection of the sulphanic acid (2110 equivalents) to the slurry, 20457 equivalents of alkaline material (18315 equivalents from the sodium tripolyphosphate and 2142 equivalents from the sodium hydroxide) were present in the slurry, that is to say, the equivalent ratio of alkali to sulphanic acid was 9.7:1, without counting the alkali present in the sodium silicate solution and minor ingredients.

The spray dried detergent powder had very good physical properties and a bulk density of 490 g/litre.

The level of undissolved material remaining after 2 minutes' stirring in water at 40°C was 0.1%. This very low result confirmed that no unusual adverse reaction had occurred between the injected sulphanic acid and the silicate in the slurry leading to the formation of high levels of insoluble siliceous material.

Claims

1. A process for the manufacture of a particulate detergent composition, which comprises the steps of:
   (i) preparing an aqueous slurry comprising one or more anionic detergent-active compounds, and
   (ii) spray-drying the slurry to form a powder;

2. A process as claimed in claim 1, wherein the anionic detergent-active compound incorporated into the slurry in wholly or predominantly acid form is an C₈-C₁₅ alkylbenzene sulphanic acid.

3. A process as claimed in claim 1 or claim 2, wherein the alkaline material incorporated in the slurry before the incorporation of the anionic detergent-active compound in wholly or predominantly acid form includes sodium hydroxide.

4. A process as claimed in any one of claims 1 to 3, wherein the alkaline material incorporated in the slurry before the incorporation of the anionic detergent-active compound in wholly or predominantly acid form includes one or more alkaline builder salts.

5. A process as claimed in claim 4, wherein the alkaline builder salts comprise sodium tripolyphosphate, sodium carbonate or a mixture thereof.

6. A process as claimed in claim 5, wherein the detergent-active compound in wholly or predominantly acid form is incorporated in the slurry after the incorporation of any sodium tripolyphosphate.

7. A process as claimed in any preceding claim, wherein the alkaline material incorporated in the slurry before the incorporation of the anionic detergent-active compound in wholly or predominantly acid form includes alkaline sodium silicate.

8. A process as claimed in any preceding claim, wherein the anionic detergent-active compound in wholly or predominantly acid form is incorporated in the slurry in an amount corresponding to 20% by weight or more of the neutralised detergent-active material in the spray-dried powder.

9. A process as claimed in any preceding claim, wherein the anionic detergent-active compound is incorporated in the slurry in 0 to 10% by weight neutralised form.

10. A process as claimed in any preceding claim, wherein the slurry is prepared in a slurry-making vessel and then pumped through pipes to a spray-drying tower, characterised in that the anionic detergent-active compound in wholly or predominantly acid form is injected into a pipe or pump between the slurry-making vessel and the spray-drying tower.

11. A process as claimed in claim 10, wherein the anionic detergent-active compound in wholly or
predominantly acid form is premixed with a liquid nonionic detergent-active compound before incorporation in the slurry.

12. A process as claimed in claim 10, wherein the anionic detergent-active compound in wholly or predominantly acid form is heated to a temperature within the range of from 50 to 60 °C before incorporation in the slurry.

**Reivendications**

1. Procédé de fabrication d'une composition détergente particulaire, qui consiste :
   (i) à préparer une bouillie aqueuse comprenant un (ou plusieurs) composé détergent actif anionique non savonneux et un (ou plusieurs) sel minéral alcalin, et
   (ii) à sécher par pulvérisation la bouillie pour former une poudre ;
   procédé dans lequel au stade (i) on incorpore un composé détergent actif anionique sous forme entièrement ou de façon prédominante acide dans la bouillie après incorporation de la matière alcaline en une proportion telle qu'un total d'au moins deux équivalents de matière alcaline par équivalent dudit détergent actif acide soit présent, à la condition que le carbonate de sodium ne soit pas utilisé comme seul sel minéral alcalin.

2. Procédé selon la revendication 1, dans lequel le composé détergent actif anionique incorporé dans la bouillie sous forme totale ou prédominante d'un acide est l'acide alkylbenzenesulfonylique en C₉₋₁₅.

3. Procédé selon la revendication 1 ou 2, dans lequel la matière alcaline incorporée dans la bouillie avant l'incorporation du détergent anionique actif sous une forme entièrement ou principalement acide contient de l'hydroxyde de sodium.

4. Procédé selon l'une quelconque des revendications 1 à 3, dans lequel la matière alcaline incorporée dans la bouillie avant l'incorporation du détergent anionique actif sous une forme entièrement ou principalement acide comprend un (ou plusieurs) sel adjuvant alcalin.

5. Procédé selon la revendication 4, dans lequel les sels adjuvants alcalins comprennent du triphosphate de sodium, du carbonate de sodium ou un mélange de ceux-ci.

6. Procédé selon la revendication 5, dans lequel le détergent actif sous forme entièrement ou principalement acide est incorporé dans la bouillie après l'introduction d'un triphosphate de sodium.

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel la matière alcaline incorporée dans la bouillie avant l'incorporation du détergent anionique sous une forme entièrement ou principalement acide contient du silicate de sodium alcalin.

8. Procédé selon l'une quelconque des revendications précédentes, dans lequel on incorpore le détergent anionique actif sous une forme entièrement ou principalement acide dans la bouillie en une quantité qui correspond à 20% en poids ou plus de la matière détergente active neutralisée dans la poudre séchée par pulvérisation.

9. Procédé selon l'une quelconque des revendications précédentes, dans lequel le détergent actif anionique est incorporé dans la bouillie sous une forme neutralisée de 0 à 10% en poids.

10. Procédé selon l'une quelconque des revendications précédentes, dans lequel on prépare la bouillie dans un récipient de production de bouillie et, ensuite, on le pompe par des conduites jusqu'à une tour de séchage par pulvérisation, caractérisé en ce que le composé détergent actif anionique sous une forme entièrement ou principalement acide est injecté dans une conduite ou une pompe entre le récipient de production de bouillie et la tour de séchage par pulvérisation.

11. Procédé selon la revendication 10, dans lequel on mélange au préalable le détergent actif anionique sous une forme entièrement ou principalement acide avec un détergent actif non ionique liquide avant l'incorporation dans la bouillie.
12. Procédé selon la revendication 10, dans lequel on chauffe le détergent actif anionique sous une forme entièrement ou principalement acide à une température de 50 à 60 °C avant de l’incorporer dans la bouillie.

5 Patentansprüche

1. Verfahren zur Herstellung einer teiltchenförmigen Detergenszusammensetzung, das die folgenden Schritte umfaßt:
   (i) Herstellen einer wässrigen Aufschlammung, die eine oder mehrere anionische Detergens-aktive Verbindungen und eines oder mehrere alkalische anorganische Salze umfaßt, und
   (ii) Sprühtrocknen der Aufschlammung um ein Pulver zu bilden, worin in Schritt (i) eine anionische Detergens-aktive Verbindung in reiner oder überwiegender Säureform der Aufschlammung nach dem Zusatz von alkalischem Material zugesetzt wird in einer Menge derart, daß eine Gesamtheit von wenigstens zwei Äquivalenten alkalischen Materials pro Äquivalent der sauren Detergens-aktiven Verbindungen anwesend sind, mit der Maßgabe, daß Natriumcarbonat nicht als das einzige alkalische anorganische Salz verwendet wird.

2. Verfahren nach Anspruch 1, worin die anionische Detergens-aktive Verbindung, die der Aufschlammung in reiner oder überwiegender Säureform zugesetzt wird, eine C_{8-15}-Alkylbenzolsulfonsäure ist.

3. Verfahren nach Anspruch 1 oder Anspruch 2, worin das alkalische Material, das der Aufschlammung vor dem Zusatz der anionischen Detergens-aktiven Verbindung in reiner oder überwiegender Säureform zugesetzt wird, Natriumhydroxid einschließt.

4. Verfahren nach einem der Ansprüche 1 bis 3, worin das alkalische Material, das der Aufschlammung vor dem Zusatz der anionischen Detergens-aktiven Verbindung in reiner oder überwiegender Säureform zugesetzt wird, eines oder mehrere alkalische Buildersalze einschließt.

5. Verfahren nach Anspruch 4, worin die alkalischen Buildersalze Natriumtripolyphosphat, Natriumcarbonat oder Mischungen davon umfassen.


7. Verfahren nach einem der vorhergehenden Ansprüche, worin das alkalische Material, das der Aufschlammung vor dem Zusatz der anionischen Detergens-aktiven Verbindung in reiner oder überwiegender Säureform zugesetzt wird, alkalisches Natriumsilikat einschließt.


9. Verfahren nach einem der vorhergehenden Ansprüche, worin die anionische Detergens-aktive Verbindung in 0 bis 10 Gew.-% neutralisierter Form zugesetzt wird.

10. Verfahren nach einem der vorhergehenden Ansprüche, worin die Aufschlammung in einem Aufschlammung-produzierenden Gefäß hergestellt wird und dann durch Röhren zu einem Sprühtrocknungsturm gepumpt wird, dadurch gekennzeichnet, daß die anionische Detergens-aktive Verbindung in reiner oder überwiegender Säureform in eine Röhre oder Pumpe zwischen dem die Aufschlammung-produzierenden Gefäß und dem Sprühtrocknungsturm eingespritzt wird.

11. Verfahren nach Anspruch 10, worin die anionische Detergens-aktive Verbindung in reiner oder überwiegender Säureform mit einer flüssigen nicht-ionischen Detergens-aktiven Verbindung vor dem Zusatz in die Aufschlammung vorgemischt wird.

12. Verfahren nach Anspruch 10, worin die anionische Detergens-aktive Verbindung in reiner oder überwiegender Säureform auf eine Temperatur im Bereich von 50 bis 80 °C vor dem Zusatz in die
Aufschlammung erhitzt wird.