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(54) **Process for preparing a detergent**

Verfahren zur Herstellung von Tensiden

Procédé de préparation de détergents

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Description

TECHNICAL FIELD

[0001] THE PRESENT INVENTION relates to cleaning compositions, and more particularly to processes for preparing granular detergent compositions having relatively high bulk density.

BACKGROUND

[0002] There has been considerable interest in the detergent industry in the development of cleaning compositions having a relatively high bulk density, typically 500 g/l and above. Such high bulk density compositions are of interest in industry as they facilitate the production of detergent compositions having a low dosage volume, with the associated conservation of resources.

[0003] In general there are two main types of process by which detergent granules or powders are prepared. The first involves spray-drying an aqueous detergent slurry in a spray-drying tower, wherein the detergent active is dried by atomising it and spraying it into a stream of air at a high temperature. There has been considerable research and development into spray-drying process for the production of detergent compositions, for example, see the processes described in EP 360275, GB 2231579, WO 99/19453 and US 4,524,010.

[0004] The second type of process involves dry mixing the detergent components, and subsequently agglomerating the dry-mixed components in a high or medium speed mixer/densifier, typically in the presence of a liquid binder, such as water, a non-ionic or an anionic surfactant.

[0005] In practice spray-dried detergent compositions have been found to have relatively low bulk density. Spray-drying processes require expensive and complicated machinery and involve relatively high energy consumption. Furthermore, spray-drying processes typically result in the production of fine particles of the detergent composition, with the associated problems with regard to air pollution.

[0006] Detergent compositions having medium to high bulk densities have been produced by dry-mix agglomeration processes. However detergents produced by such dry-mix processes have been found to suffer from a number of problems, including poor dispersion properties and unsatisfactory detergent performance, as discussed in US 6,303,558. Dry-mix processes also require the use of hi-tech mixer/densifier equipment.

[0007] Accordingly, there remains a need for alternative processes for the production of granular cleaning compositions having a relatively high bulk density. Also, there remains a need for such a process which is efficient and economical to facilitate large-scale production of granular cleaning compositions having a relatively high bulk density.

[0008] The inventors of the present Application have found that granular cleaning compositions having a relatively high bulk density may be prepared by a process involving adding water-soluble salts to an aqueous solution of sodium laurel sulphate. The process disclosed herein allows the preparation of a granular cleaning composition without the requirement for expensive spray-drying or mixer/densifier equipment.

SUMMARY OF THE INVENTION

[0009] According to the present invention there is provided a process for the production of a high bulk density granular detergent composition or component having a bulk density of at least 500 g/l, comprising the steps of;

(i) providing an aqueous solution of sodium laurel sulphate,
(ii) adding one or more water-soluble salts to the aqueous sodium laurel sulphate solution to precipitate the sodium laurel sulphate,
(iii) separating the resulting precipitate, and, finally;
(iv) drying the product.

[0010] Conveniently at least one water-soluble salt is chosen from the group consisting of potassium nitrate, potassium chloride, potassium acetate, sodium acetate, sodium chloride, barium chloride, or any combination thereof.

[0011] Preferably at least one water-soluble salt is a sea salt.

[0012] Conveniently the separation of the precipitate in step (iii) is effected with a filtration apparatus or centrifuge.

[0013] Preferably the separation of the precipitate is effected with a filter press.

[0014] Conveniently the product may be dried in step (iv) in a fluid bed dryer, drum vacuum dryer, tray dryer, or any combination thereof.

[0015] According to one aspect of the present invention one or more additives may be advantageously added to the separated precipitate formed in step (iii) before drying, to form detergents or cleaning compositions having desired properties.

[0016] Conveniently one or more of the additives are chosen from the group consisting of detergency builders, fillers, acid powders, alkali powders, binders, bleaches, bleach activators, fluorescers, anti-tarnish agents, anti-corrosion agents, soil-suspending agents, soil-release agents, germicides, Ph adjusting agents, chelating agent, clays, coating agents, enzymes, enzyme stabilising agents or any combination thereof.

[0017] Preferred additives include acid powders and alkali powders.

[0018] The process described herein allows the preparation of relatively high bulk density granular surfactant, detergent or cleaning compositions, without the need for expensive spray-drying, or mixer/densifier equipment.

DETAILED DESCRIPTION OF THE INVENTION

[0019] The present invention is concerned with the preparation of a relatively high bulk density detergent composition or component by means of a precipitation process which involves the addition of a water-soluble salt to an aqueous sodium laurel sulphate solution.

[0020] Sodium Laurel Sulphate (SLS) is widely used in the detergent industry as an active component in a wide variety of cleaning compositions, such as, for example, washing powders, soap based products and shampoos.

[0021] The aqueous SLS solution according to the process of the present invention preferably has a concentration of 5 to 40% (w/v),preferably 10 to 35% (w/v) and more preferably 20 to 30% (w/v). The aqueous SLS solution for use in the present process may be prepared by any suitable method. Suitable methods for the preparation of the aqueous SLS solution will be evident to the person skilled in the art, with respect to the particular surfactant selected.

[0022] The aqueous SLS solution may conveniently, be prepared by a sulphonation process. Suitable conditions for carrying out such a sulphonation process will be known to the person skilled in the art. The sulphonation process may be either a continuous or batch type processes. Continuous sulphonation processes will typically produce aqueous solution of Sodium Laurel Sulphate with a concentration of around 70% (w/v). Batch processes will typically produce an aqueous solutions with a lower concentration of Sodium Laurel Sulphate, typically around 30% to 60% (w/v). Concentrated aqueous solution of Sodium Laurel Sulphate produced in this way can then be diluted to the desired concentration for use in the present process. The concentrated aqueous solution of Sodium Laurel Sulphate may be prepared in-situ, or is readily available from manufacturers. Alternative methods for the production of aqueous solution of Sodium Laurel Sulphate will be evident to the person skilled in the art.

[0023] In the second step of the process one or more water-soluble salts are added to the aqueous surfactant solution to induce precipitation. Salts contemplated for use in this step include organic and inorganic salts.

[0024] The amount of salt to be added to the aqueous solution of SLS must be an amount sufficient to effect precipitation of the SLS from solution. The amount of salt required to effect precipitation in any given case can be easily determined be the person skilled in the art using standard procedures.

[0025] Specifically, it has been observed that the surfactant Sodium Laurel Sulphate is highly soluble in water, but only poorly soluble in aqueous salt solution. It has been found that increasing the concentration of salt in the aqueous solution results in decrease in the solubility of the surfactant compound. With respect to the surfactant Sodium Laurel Sulphate, it has been observed that Sodium Laurel Sulphate is slightly soluble in an aqueous salt solution having a salt concentration of up to about 3% (w/v), and is progressively more insoluble on increase of the salt concentration. It has been observed that Sodium Laurel Sulphate is almost completely insoluble in an aqueous salt solution having a salt concentration of 5% (w/v). The amount of salt added may usefully be an amount to provide aqueous solution having a salt concentration of 5% (w/v) or more, preferably 10% (w/v) or more.

[0026] It is important that sufficient salt be added in order to induce precipitation. If sufficient salt is not added separation may not be achieved and a paste may be formed comprising surfactant and salt components.

[0027] The salt may conveniently be added to the aqueous solution of SLS at room temperature. Preferably the aqueous solution is mixed or agitated on addition of the salt.

[0028] Preferred salts for use in the process include potassium nitrate, potassium chloride, barium chloride, sodium chloride, sodium acetate, potassium acetate or combinations thereof. It has been found that variation of the salt added to the aqueous solution of Sodium Laurel Sulphate, results in variations in the physical characteristics, e.g. density, particle size, shape and form of the resultant granular product.

[0029] One or more salts may be used to obtain desired particle size, density, shape and form.

[0030] Particularly preferred salts for use in the process include salts from natural sea water. Sea salts contain a mixture of natural salts, the major component being sodium chloride. It has been found that granular product having a bulk density of over 500 g/l can be achieved using sea salts. Sea salts have the advantages of being cheap and readily available by evaporation of sea water.

[0031] The particular method used for separating the surfactant precipitate is not important. Any simple separation method may conveniently be used, for example, filtration methods e.g. filter press, vacuum filtration, or with a centrifuge. The use of a filter press is particularly suitable for producing a separated precipitate having a low moisture content.

[0032] On separation a wet cake of the SLS precipitate is formed. The separated precipitate preferably has a moisture

content of 5% to 35% (w/w).

[0033] If desired, after separation of the SLS precipitate, the aqueous salt solution can be concentrated and the salts recovered by known methods. For example, simple solar evaporation methods can be conveniently be used for recovery of many salts, particularly where sea salts are used. In this way the salt solution by-product can be recycled, with the associated economic and environmental advantages.

[0034] Optionally one or more additives may be added to the separated SLS precipitate before drying.

[0035] Of particular interest are high density powders, liquid additives or fillers which can be added to produce detergent compositions having a higher bulk density than dried granules of surfactant composition alone, for instance to obtain granules with a high bulk density of over 700g/l, and/or to provide variation in the particle size, form or shape of the resultant granular detergent product.

[0036] Also acidic powders, such as Picric acid, para toluene sulphonic acid and/or alkaline powders, such as sodium carbonate, or sodium bicarbonate, can be added to enhance the dissolution and foaming properties of the detergent composition. It has been found that, where a mixture of alkaline and acidic powders are added, as additives, the product detergent composition liberates carbon dioxide gas in aqueous media, improving the solubility and dispersion properties of the detergent composition.

[0037] In general, any standard detergent additives can be added to provide granular detergent composition having desired properties. Preferred additives include fillers, detergency, builders, acid powders, alkali powders, binders, bleaches, bleach activators, fluorescer anti-tarnish agents, anti-corrosion agents, soil-suspending agents, soil-release agents, germicides, Ph adjusting agents, chelating agents, clays, coating agents, enzymes, enzyme stabilising agents or any combination thereof. Other suitable additives will be well known to the person skilled in the art.

[0038] Drying of the product in step (iv) can be carried out in any simple dryer. Examples of suitable simple dryers include fluid bed dryers, tray dryers or drum vacuum dryers. Other suitable dryers will be known to the skilled person. It has been found that granular product having a moisture content in the range of 0.5%-2% (w/w) can be achieved using a vacuum dryer.

[0039] Drying the wet-cake of surfactant in this manner requires a considerable lower energy consumption than that used in many conventional processes for the drying of surfactant slurry or aqueous solution (e.g spray-dry methods).

[0040] The process according to the present invention allows the production of high bulk density granular free-flowing detergent component or composition having a bulk density of at least 500 g/l. The process of the invention provides an economic and efficient method for the preparation of high bulk density, free-flowing granular detergent compositions. The process allows the production of granular products having a range of desired bulk densities, particle sizes, shapes and forms, which can be used in a variety of detergent and cleaning composition applications.

[0041] The use of the method of the present invention for the manufacture of granular detergent compositions allows the production of fine particles of detergent composition to be avoided, with the associated safety and environmental advantages.

[0042] The process of the invention can be used to provide granules of Sodium Laurel Sulphate having a bulk density in the range of between 500 to 710 g/l.

[0043] The process of the present invention avoids the need for complicated and expensive equipment, and provides an economical and energy-efficient process for the manufacture of low dosage volume, compact granular surfactant and detergent compositions. Additionally the aqueous salt solution by-product produced in the process of the present invention as described can be recycled, recovering the corresponding salts.

[0044] The invention is further illustrated by the following non-limiting examples.

EXAMPLES

Example 1

[0045] To 100 ml of a 20% (w/v) solution of Sodium Laurel Sulphate at room temperature 10g of barium chloride were added with continuous stirring. The mixture was agitated for two hours. Precipitated granules of Sodium Laurel Sulphate were observed at the bottom of the vessel. The precipitate was filtered under vacuum, producing a wet cake of Sodium Laurel Sulphate. The wet cake of Sodium Laurel Sulphate was dried under vacuum and the properties of the resultant Sodium Laurel Sulphate granules were determined as follows:-

Density	710 g/l (measured using standard tap density apparatus)
Particle size	100 micron - 50%
	50 micron - 20%
	30 micron - 30%
Shape/form	rounded/globular granules

(continued)

(Particle size determined by visual microscopic method)

Examples 2 to 6

[0046] The process was carried out as in Example 1 with the addition of the different salts as shown in Table 1 below to 100ml of 20% (w/v) solution of Sodium Laurel Sulphate:

Salt (10g)	Bulk density of granules (g/l)
Potassium nitrate	700
Potassium chloride	680
Potassium acetate	620
Sodium acetate	590
Sodium chloride	550

Claims

1. A process for preparing a granular detergent composition or component having a bulk density of at least 500 g/l comprising the steps of;

   (i) providing an aqueous solution of sodium laurel sulphate,
   (ii) adding one or more water-soluble salts to the aqueous sodium laurel sulphate solution to precipitate the sodium laurel sulphate,
   (iii) separating the resulting precipitate, and, finally;
   (iv) drying the product.

2. A process according to Claim 1, wherein at least one water-soluble salt is chosen from the group consisting of potassium nitrate, potassium chloride, potassium acetate, sodium acetate, sodium chloride, or any combination thereof.

3. A process according to any one of the preceding claims wherein at least one water-soluble salt is a sea salt.

4. A process according to any one of the preceding claims wherein the precipitate is separated in step (iii) with a filtration apparatus or centrifuge.

5. A process according to any one of the preceding claims wherein the product is dried in step (iv) in a fluid bed dryer, drum vacuum dryer, tray dryer, or any combination thereof.

6. A process according to any one of the preceding claims further comprising the step of adding one or more additives to the separated precipitate before drying.

7. A process according to Claim 6, wherein the one or more additives are chosen from the group consisting of detergency builders, fillers, acid powders, alkali powders, binders, bleaches, bleach activators, fluorescers, anti-tarnish agents, anti-corrosion agents, soil-suspending agents, soil-release agents, germicides, Ph adjusting agents, chelating agents, clays, coating agents, enzymes, enzyme stabilising agents or any combination thereof.

8. A process according to Claim 7, wherein the additives include an acid powder and an alkali powder.

Patentansprüche

1. Verfahren zur Herstellung einer körnigen Reinigungsmittelzusammensetzung oder komponente mit einer Schüttdichte von wenigstens 500 g/l, das die Schritte umfasst:

(i) Bereitstellen einer wässrigen Lösung von Natriumlaurelsulfat,

(ii) Zugeben eines oder mehrerer wasserlöslicher Salze zur wässrigen Natriumlaurelsulfat-Lösung, um das Natriumlaurelsulfat auszufällen,

(iii) Abtrennen des resultierenden Niederschlags und schließlich

(iv) Trocknen des Produktes.

2. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, daß** wenigstens ein wasserlösliches Salz ausgewählt wird aus der Gruppe, die aus Kaliumnitrat, Kaliumchlorid, Kaliumacetat, Natriumacetat, Natriumchlorid oder irgendeiner Kombination davon besteht.

3. Verfahren nach einem der vorangehenden Ansprüche, **dadurch gekennzeichnet, daß** wenigstens ein wasserlösliches Salz ein Meersalz ist.

4. Verfahren nach einem der vorangehenden Ansprüche, **dadurch gekennzeichnet, daß** der Niederschlag in Schritt (iii) mit einer Filtrationsvorrichtung oder Zentrifuge abgetrennt wird.

5. Verfahren nach einem der vorangehenden Ansprüche, **dadurch gekennzeichnet, daß** das Produkt in Schritt (iv) in einem Fließbetttrockner, Trommelvakuumtrockner, Hordentrockner oder irgendeiner Kombination davon getrocknet wird.

6. Verfahren nach einem der vorangehenden Ansprüche, **dadurch gekennzeichnet, daß** es weiter den Schritt umfasst, daß ein oder mehrere Zusatzstoffe zum abgetrennten Niederschlag vor dem Trocknen hinzugefügt werden.

7. Verfahren nach Anspruch 6, **dadurch gekennzeichnet, daß** der eine oder die mehreren Zusatzstoffe ausgewählt werden aus der Gruppe, die aus Reinigungsmittel-Buildern, Füllstoffen, sauren Pulvern, alkalischen Pulvern, Bindemitteln, Bleichmitteln, Bleichaktivatoren, Aufhellern, Mitteln zur Verhinderung des Anlaufens, Mitteln zur Verhinderung der Korrosion, Mitteln zum Suspendieren von Schmutz, Mitteln zum Freisetzen von Schmutz, Germiziden, pH-Einstellungsmitteln, Chelatbildnern, Tonen, Beschichtungsmitteln, Enzymen, Enzymstabilisierungsmitteln oder irgendeiner Kombination davon besteht.

8. Verfahren nach Anspruch 7, **dadurch gekennzeichnet, daß** die Zusatzstoffe ein saures Pulver und ein alkalisches Pulver einschließen.

Revendications

1. Un procédé pour préparer une composition ou un composant détergent granulaire ayant une masse spécifique d'au moins 500 g/l comprenant les étapes consistant à :

(i) former une solution aqueuse de sodium laurel sulfate,
(ii) ajouter un ou plusieurs sels solubles dans l'eau à la solution aqueuse de sodium laurel sulfate pour précipiter le sodium laurel sulfate,
(iii) séparer le précipité résultant,
et, finalement,
(iv) sécher le produit.

2. Un procédé selon la revendication 1, dans lequel au moins un sel soluble dans l'eau est choisi dans le groupe comprenant le nitrate de potassium, le chlorure de potassium, l'acétate de potassium, l'acétate de sodium, le chlorure de sodium ou n'importe quelle combinaison de ceux-ci.

3. Un procédé selon l'une quelconque des revendications précédentes dans lequel au moins un sel soluble dans l'eau est le sel marin.

4. Un procédé selon l'une quelconque des revendications précédentes dans lequel, dans l'étape (iii), le précipité est séparé par un appareil de filtration ou de centrifugation.

5. Un procédé selon l'une quelconque des revendications précédentes dans lequel, dans l'étape (iv), le produit est séché dans un séchoir à lit fluide, un séchoir à tambour sous vide, un séchoir à plateau ou n'importe quelle combinaison de ceux-ci.

6. Un procédé selon l'une quelconque des revendications précédentes comprenant de plus l'étape consistant à ajouter un ou plusieurs additifs au précipité séparé, avant séchage.

7. Un procédé selon la revendication 6, dans lequel un ou plusieurs additifs sont choisis dans le groupe comprenant des agents de détergence, des charges, des poudres acides, des poudres alcalines, des liants, des agents de blanchiment, des activateurs d'agents de blanchiment, des agents de fluorescence, des agents anti-ternissure, des agents anti-corrosion, des agents de mise en suspension de souillures, des agents d'élimination de souillures, des germicides, des agents d'ajustement du pH, des agents complexants, des argiles, des agents d'enduction, des enzymes, des agents de stabilisation d'enzyme ou n'importe quelle combinaison de ceux-ci.

8. Un procédé selon la revendication 7, dans lequel les additifs comportent une poudre acide et une poudre alcaline.