

(19)



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11) Publication number:

0 262 896 B1

(12)

EUROPEAN PATENT SPECIFICATION(45) Date of publication of patent specification: **18.03.92** (51) Int. Cl.⁵: **C11D 9/26**, C11D 17/06(21) Application number: **87308556.7**(22) Date of filing: **28.09.87**

The file contains technical information submitted
after the application was filed and not included in
this specification

(54) **Detergent flakes and process for their production.**(30) Priority: **30.09.86 GB 8623426**(43) Date of publication of application:
06.04.88 Bulletin 88/14(45) Publication of the grant of the patent:
18.03.92 Bulletin 92/12(84) Designated Contracting States:
CH DE ES FR GB IT LI NL SE(56) References cited:
DE-A- 2 041 794
NL-A- 6 715 965
US-A- 1 942 418
US-A- 2 295 596
US-A- 2 429 852(73) Proprietor: **UNILEVER PLC**
Unilever House Blackfriars P.O. Box 68
London EC4P 4BO(GB)(84) Designated Contracting States:
GB(73) Proprietor: **UNILEVER NV**
Burgemeester s'Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam(NL)(84) Designated Contracting States:
CH DE ES FR IT LI NL SE(72) Inventor: **Irvin, Carl Martin**
51 Heathbank Avenue
Irby Wirral(GB)(74) Representative: **Thomas, Susan Margaret et al**
UNILEVER PLC Patents Division P.O. Box 68
Unilever House
London EC4P 4BO(GB)**EP 0 262 896 B1**

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

The present invention relates to detergent flakes and to a process for making detergent flakes.

Detergent flakes are intended primarily for laundry uses. They are particularly appropriate for laundering delicate and fine fabrics at a relatively low temperature, for example between 20 and 40°C. Typically detergent flakes are employed when washing a small quantity of laundry by hand. It is therefore a necessary requirement of detergent flakes that they dissolve relatively readily in the low temperature washing water employed.

US-A-1 942 418 (Lever Brothers Co) discloses a novel process for the manufacture of soap flakes, such that the flakes are of the approximate dimensions of one quarter inch in length (6.25mm) by 0.002 inch in thickness (0.05mm).

US-A-2 295 596 (Procter & Gamble Co) discloses a soap flake product containing a substantial proportion of sodium soap in the beta phase, which usually has a moisture content of 3-15%.

DE-A-2 041 794 (Procter & Gamble Co) and NL-A-6715965 (Procter & Gamble Co) disclose toilet soap bar compositions comprising alkali metal soaps (usually sodium) of a fatty acid mixture, the soap being incompletely neutralised such that a minor amount of free fatty acid is present.

According to a first aspect of the present invention there are provided cut detergent flakes having a thickness between 0.05mm and 0.2mm and comprising 80 to 90wt% soap calculated on an anhydrous basis, 1 to 10wt% free fatty acid and 6 to 12wt% moisture.

The present invention can thus provide cut detergent flakes whose major components are derived wholly from soap and/or soap-making sources. Minor components that can additionally be present include sequestrants, anti-oxidants, preservatives, perfumes and dyes.

The detergent flakes embodying the present invention have a substantially constant thickness within the range 0.05 to 0.2mm, preferably 0.05mm to 0.1mm. The overall dimensions of the flakes are determined primarily by the requirement that the flakes dissolve within a relative short space of time. Preferably the individual total perimeter dimension of each flake is between 20mm and 50mm. Suitably the flakes are diamond shaped. Other shapes and sizes can however be envisaged.

The presence of the free fatty acid in the present flakes can improve the solubility of the flakes compared to equivalent flakes not containing free fatty acid. The increased solubility in terms of time taken to achieve substantially full dissolution can be particularly noticeable at temperatures of 30°C and below. The present invention can additionally allow good solubility to be achieved when the soap component of the flakes is derived solely from cheap and readily available sources such as a major proportion of tallow and a minor proportion of coconut oil.

Preferably the free fatty acid is derived wholly from soap and/or soap-making sources. The major components of the present detergent flakes can thus be derived wholly from natural renewable resources. The presence of the free fatty acid can moreover render the washing solution less irritant to the skin. The added free fatty acid can, but need not necessarily, be distilled free fatty acid. The flakes can contain a dye, but in the absence of any added colouring matter the flakes can have a good white colour.

The detergent flakes preferably comprise 84 to 88wt% soap, 3 to 10wt% free fatty acid and 6 to 11wt% moisture, more preferably 85 to 87wt% soap, 3 to 5wt% free fatty acid and 8 to 10wt% moisture. The precise composition will depend on the actual ingredients employed, but must be within the presently defined limits in order for the composition to be in cut flake form. The free fatty acid is calculated with respect to the actual quantities of free fatty acid present in the end product, which may be less than, for example, the amount of distilled fatty acid added, but would include free fatty acid already present in the soap components of the composition.

Sources for the soap can be any conventional soap-making material. Preferably however they are derived from renewable natural resources such as triglyceride fats and oils. A preferred composition includes a tallow or tallow-like material combined with a lauric-containing material. Suitably the soap component comprises 40 to 95wt% tallow soap and 60 to 5wt% coconut soap, more suitably 60 to 95wt% tallow soap and 40 to 5wt% coconut soap, even more suitably 75 to 90wt% tallow soap and 25 to 10wt% coconut soap.

The fatty acid is preferably derived from a triglyceride oil or fat. Suitably it is coconut or palm kernel or a tallow/coconut mixture of fatty acids.

According to a second aspect of the present invention there is provided a process for making detergent flakes comprising rolling into sheet form a composition comprising 80 to 90wt% soap calculated on an anhydrous basis, 1 to 10wt% free fatty acid and 6 to 12wt% moisture to a thickness between 0.05mm and 0.2mm and cutting the resulting sheet into flakes.

The composition has the preferred compositional ranges and sources mentioned above. Preferably the

composition is rolled and cut into flakes having a thickness between 0.05mm and 0.1mm.

By use of the present process it has been found possible to make cut flakes of the present thickness containing free fatty acid. It is essential that the present compositional ranges are employed. If the composition contains too much moisture and/or free fatty acid, the resulting sheet will be difficult to roll and too sticky to form into discrete flakes and if too little moisture and/or free fatty acid it will be too dry and fragile to form flakes. The precise composition selected will depend on the ingredients and more importantly on the process conditions and the actual design of the rolling mill employed. For example, a multi roll mill or single roll mill can be employed.

Suitably the present process includes blending the free fatty acid with a neat soap solution containing the appropriate soap ingredients. Minor ingredients such as sequestrants, preservatives and anti-oxidants can be conveniently added at this stage. The resulting molten mixture at a temperature between 100 and 160°C can then be preferably dried to a moisture content between 6 and 12wt% using for example a vacuum drier or alternatively an expansion chamber/chilled drum. The composition can be removed from the drier and, optionally after an ageing step, it can then be noodled and can be stored until required. Any perfume can be added prior to an optional further noodling step and milling. Suitably a three or a five roll mill can be employed. The rolling step suitably takes place with the composition at a temperature between 20 and 30°C. The resulting sheet is cut into flakes and the flakes can be ready for packing. The cutting action can be full thickness through the sheet or to a partial depth sufficient in a subsequent step of stripping the flakes from the end roller by means of a doctor blade to cause the sheet to break crisply into discrete flakes of the intended shape. The flakes can be cut into any desired size and shape. A suitable size is flakes having individual total perimeter between 20mm and 50mm. A suitable shape is diamond shaped, but other shapes can be chosen.

Embodiments of the present invention will now be described by way of example only with reference to the following Examples.

Example 1

A blend of 82wt% high grade tallow and 18wt% coconut oil was saponified using sodium hydroxide and steam. The neat soap, containing 63wt% total fatty matter, was separated from the glycerine by washing with brine.

4.7wt%, based on the weight of the final product, of distilled free fatty acid (Prifac 7970, Trade Mark) and small amounts of conventional sequestrant, anti-oxidants and perfume were admixed with the neat soap. 4.7 wt% distilled free fatty acid is equivalent to 3.9 wt% free fatty acid in the final product. The resulting mixture was dried to reduce its water content from about 30wt% to about 9wt%. Perfume was added to the composition which was then noodled and roll milled to the desired thickness of 0.05 to 0.1mm. The resulting thin sheet was cut into diamond shaped flakes having a side length of 5mm, collected and packed. The resulting flakes had a final composition of 86wt% soap and 8.8wt% moisture (including salt and glycerol) plus the ingredients mentioned above.

Example 2

The procedure of Example 1 was followed with the difference that 3.6 wt%, based on the weight of the final product, of distilled free fatty acid (Prifac 7970, Trade Mark) were employed in place of the 4.7 wt% used in Example 1. 3.6 wt% distilled free fatty acid is equivalent to 3.0 wt% free fatty acid in the final product.

The product flakes having a thickness between 0.05 and 0.1 mm were readily formed by rolling and cutting. The flakes had a final composition of 87 wt% soap and 8.9 wt% moisture (including salt and glycerol) plus the free fatty acid and added sesquestant, anti-oxidant and perfume.

Tests were performed on the product flakes of Examples 1 and 2, on equivalent flakes (B) not containing distilled fatty acid, and for further comparison on two commercially available soap flake products (C and D) based on soap and moisture only as their main ingredients.

Each test was performed twice on each product and the average of the results taken. Each test comprised placing 400cm³ water of 26°FH in a standard beaker and stabilising the water temperature at either 30°C or 40°C. The water was stirred by a mechanical stirrer to create a vortex and 4g of test product was poured into the vortex. The solution was stirred at a fixed rate for 2 minutes and was then filtered so as to allow the undissolved residues of each product to be collected and weighed and hence to yield a figure for the insolubility of each product. Each product was tested at 30°C and 40°C. The results are given in the Table below.

Table

Product	wt% insoluble at 30 ° C	wt% insoluble at 40 ° C
Example 1	0.70	0.20
Example 2	2.20	0.20
Comparative product B	45.20	0.44
Commercial product C	57.24	7.19
Commercial product D	42.72	0.67

As can be seen from the above Table, the flakes of Examples 1 and 2 embodying the present invention showed significantly better solubility, especially at 30 ° C than the comparative test products.

Claims

1. Cut detergent flakes having a thickness between 0.05mm and 0.2mm and comprising 80 to 90wt% soap calculated on an anhydrous basis, 1 to 10wt% free fatty acid and 6 to 12wt% moisture.
2. Detergent flakes according to claim 1 comprising 84 to 88wt% soap, 3 to 10wt% free fatty acid and 6 to 11wt% moisture.
3. Detergent flakes according to claim 2 comprising 85 to 87 wt% soap, 3 to 5wt% free fatty acid and 8 to 10 wt% moisture.
4. Detergent flakes according to any one of claims 1 to 3 wherein the soap component comprises 40 to 95wt% tallow soap and 60 to 5wt% coconut soap, calculated with respect to the soap content.
5. Detergent flakes according to claim 4 wherein the soap component comprises 60 to 95wt% tallow soap and 40 to 5wt% coconut soap.
6. Detergent flakes according to any one of the preceding claims wherein the free fatty acid is a distilled fatty acid derived from triglyceride oil or fat.
7. Process for making detergent flakes comprising rolling into sheet form a composition comprising 80 to 90wt% soap calculated on an anhydrous basis, 1 to 10wt% free fatty acid and 6 to 12wt% moisture to a thickness between 0.05mm and 0.2mm and cutting the resulting sheet into flakes.
8. Process according to claim 7 wherein the composition comprises about 84 to 88wt% soap, 3 to 10wt% free fatty acid and 6 to 11wt% moisture.
9. Process according to claim 8 wherein the composition comprises 85 to 87 wt% soap, 3 to 5 wt% free fatty acid and 8 to 10 wt% moisture.
10. Process according to any one of claims 7 to 9 comprising admixing free fatty acid with a neat soap solution, drying the resulting solution and noodling the resulting composition prior to rolling the composition into sheet form.
11. A process according to any one of claims 7 to 10 wherein the composition is rolled at a temperature between 20 and 30 ° C.

Revendications

1. Détergent en paillettes découpées ayant une épaisseur comprise entre 0,05 mm et 0,2 mm et comprenant 80 à 90% en masse de savon calculé sur une base anhydre, 1 à 10% en masse d'acide gras libre et 6 à 12% en masse d'humidité.
2. Le détergent en paillettes selon la revendication 1, comprenant 84 à 88% en masse de savon, 3 à 10% en masse d'acide gras libre et 6 à 11% en masse d'humidité.

3. Le détergent en paillettes selon la revendication 2, comprenant 85 à 87% en masse de savon, 3 à 5% en masse d'acide gras libre et 8 à 10% en masse d'humidité.
- 5 4. Le détergent en paillettes selon l'une des revendications 1 à 3, dans lequel le composant savon comprend 40 à 95% en masse de savon de suif et 60 à 5% en masse de savon de noix de coco, calculé par rapport à la teneur en savon.
- 10 5. Le détergent en paillettes selon la revendication 4, dans lequel le composant savon comprend 60 à 95% en masse de savon de suif et 40 à 5% en masse de savon de noix de coco.
- 15 6. Le détergent en paillettes selon l'une des revendications précédentes, dans lequel l'acide gras libre est un acide distillé dérivé d'huile ou de matière grasse de triglycéride.
- 20 7. Procédé de fabrication de détergent en paillettes comprenant le laminage sous forme de feuille d'une composition comprenant 80 à 90% en masse de savon calculé sur une base anhydre, 1 à 10% en masse d'acide gras libre et 6 à 12% en masse d'humidité à une épaisseur comprise entre 0,05 mm et 0,2 mm et le découpage de la feuille qui en résulte en paillettes.
- 25 8. Le procédé selon la revendication 7, dans lequel la composition comprend environ 84 à 88% en masse de savon, 3 à 10% en masse d'acide gras libre et 6 à 11% en masse d'humidité.
- 30 9. Le procédé selon la revendication 8, dans lequel la composition comprend 85 à 87% en masse de savon, 3 à 5% en masse d'acide gras libre et 8 à 10% en masse d'humidité.
- 35 10. Le procédé selon l'une des revendications 7 à 9 comprenant l'adjonction d'acide gras libre à une solution de savon pur, le séchage de la solution qui en résulte et la découpe sous forme de nouilles de la composition qui en résulte avant le laminage de la composition sous forme de feuille.
- 40 11. Un procédé selon l'une des revendications 7 à 10, dans lequel la composition est laminée à une température comprise entre 20 et 30 ° C.

Patentansprüche

- 35 1. Geschnittene Reinigungsmittel-Flocken, die eine Dicke zwischen 0,05 mm und 0.2 mm aufweisen, und 80 bis 90 Gew.-% Seife, berechnet auf einer wasserfreien Basis, 1 bis 10 Gew.-% freie Fettsäure und 6 bis 12 Gew.-% Feuchtigkeit umfassen.
- 40 2. Reinigungsmittel-Flocken nach Anspruch 1, die 84 bis 88 Gew.-% Seife, 3 bis 10 Gew.-% freie Fettsäure und 6 bis 11 Gew.-% Feuchtigkeit umfassen.
- 45 3. Reinigungsmittel-Flocken nach Anspruch 2, die 85 bis 87 Gew.-% Seife, 3 bis 5 Gew.-% freie Fettsäure und 8 bis 10 Gew.-% Feuchtigkeit umfassen.
- 50 4. Reinigungsmittel-Flocken nach einem der Ansprüche 1 bis 3, worin die Seifen-Komponente 40 bis 95 Gew.-% Talgseife und 60 bis 5 Gew.-% Kokosnuß-Seife, bezogen auf den Seifengehalt, umfaßt.
- 55 5. Reinigungsmittel-Flocken nach Anspruch 4, worin die Seifen-Komponente 60 bis 95 Gew.-% Talgseife und 40 bis 5 Gew.-% Kokosnuß-Seife umfaßt.
- 60 6. Reinigungsmittel-Flocken nach einem der vorhergehenden Ansprüche, worin die freie Fettsäure eine destillierte Fettsäure, abgeleitet von Triglyceridöl oder Fett, ist.
- 65 7. Verfahren zur Herstellung von Reinigungsmittel-Flocken, das das Walzen in Blattform einer Zusammensetzung, die 80 bis 90 Gew.-% Seife, berechnet auf einer wasserfreien Basis, 1 bis 10 Gew.-% freie Fettsäure und 6 bis 12 Gew.-% Feuchtigkeit umfaßt, auf eine Dicke zwischen 0,05 mm und 0,2 mm und Schneiden des resultierenden Blattes in Flocken umfaßt.
- 70 8. Verfahren nach Anspruch 7, worin die Zusammensetzung 84 bis 88 Gew.-% Seife, 3 bis 10 Gew.-%

freie Fettsäure und 6 bis 11 Gew.-% Feuchtigkeit umfaßt.

9. Verfahren nach Anspruch 8, worin die Zusammensetzung 85 bis 87 Gew.-% Seife, 3 bis 5 Gew.-% freie Fettsäure und 8 bis 10 Gew.-% Feuchtigkeit umfaßt.

5

10. Verfahren nach einem der Ansprüche 7 bis 9, das das Zumischen von freier Fettsäure zu einer reinen Seifenlösung, Trocknen der resultierenden Lösung und Nudeln der resultierenden Zusammensetzung vor dem Walzen der Zusammensetzung in Blattform umfaßt.

- 10 11. Verfahren nach einem der Ansprüche 7 bis 10, worin die Zusammensetzung bei einer Temperatur zwischen 20 und 30 ° C gewalzt wird.

15

20

25

30

35

40

45

50

55