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## COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

## APPLICATION FOR A STANDARD PATENT

I\We,

UNILEVER PLC

of

UNILEVER HOUSE  
BLACKFRIARS  
LONDON EC4  
ENGLAND

hereby apply for the grant of a standard patent for an  
invention entitled:

PROCESS FOR PREPARING SOAP-ACYL ISETHIONATE  
COMPOSITIONS.

which is described in the accompanying complete specification

Details of basic application(s):

| Number of basic<br>application | Name of Convention country in<br>which basic application was<br>filed | Date of basic<br>application |
|--------------------------------|-----------------------------------------------------------------------|------------------------------|
|--------------------------------|-----------------------------------------------------------------------|------------------------------|

My/our address for service is care of ~~GRIFFITH HACK & CO.,~~  
~~Patent Attorneys, 601 St. Kilda Road, Melbourne 3004,~~  
~~Victoria, Australia.~~ UNILEVER AUSTRALIA LIMITED

ct Mr B.F. Jones

Private Bag 2, Epping NSW 2121

DATED this 08th day of April 1991

UNILEVER PLC

GRIFFITH HACK & CO.

*[Signature]*



ADDRESS FOR SERVICE  
ALTERED

TO: The Commissioner of Patents.

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AUSTRALIA

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PATENTS ACT 1952

APPLICATION  
BY ASSIGNEE  
OF INVENTOR

DECLARATION IN SUPPORT OF AN APPLICATION No. 74125/91  
FOR A PATENT

In support of an application made by: UNILEVER PLC  
for a patent for an invention entitled:

PROCESS FOR PREPARING SOAP-ACYL ISETHIONATE COMPOSITIONS  
I, Peter Geoffrey Mole  
of Unilever House, Blackfriars, London EC4, England,

do solemnly and sincerely declare as follows:

1. I am authorised by the above mentioned applicant for the patent to make this declaration on its behalf.
2. The name and address of each actual inventor of the invention is as follows:

Yuriy Osyp KUTNY, 15 St. Clair Avenue, Rutherford, New Jersey 07070, United States of America;  
Frederick Silvio OSMER, 5 Citroen Way, Parsippany, New Jersey 07054, United States of America;  
Joseph James PODGORSKY, 9 Woodward Road, Slate Hill, New York, United States of America;  
David Andrew RICHARDSON, 44 Cedar Trail, Ogden Dunes, Portage, Indiana 46368, United States of America; and  
Karla Jean RYS, 319 Division Avenue, Hasbrouck Heights, New Jersey 07604, United States of America,

3. The facts upon which the applicant is entitled to make this application are as follows:

The applicants are assignees of Lever Brothers Company, who are the assignees of the actual inventors.

Declared at London, England  
this 4th day of April 1991  
Signed P. Mole  
Position: Authorised Signatory

GRIFFITH HACK & CO  
PATENT AND TRADE MARK ATTORNEYS  
MELBOURNE . SYDNEY . PERTH



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- (71) Applicant(s)  
UNILEVER PLC
- (72) Inventor(s)  
YURIY OSYP KUTNY; FREDERICK SILVIO OSMER; JOSEPH JAMES PODGORSKY; DAVID  
ANDREW RICHARDSON; KARLA JEAN RYS
- (56) Prior Art Documents  
US 4707238  
US 4695395  
US 4663070
- (57) Claim

1. A process for the production of a detergent composition suitable for shaping into toilet bars, comprising alkali metal salts of  $C_8-C_{22}$  alkyl fatty monocarboxylic acid and  $C_8-C_{22}$  acyl isethionate in a weight ratio of 20:1 to 1:0.98, said process comprising the steps of:

(i) forming an aqueous caustic solution, at an elevated temperature, comprising sodium hydroxide and alkali metal isethionate;

(ii) charging  $C_8-C_{22}$  alkyl fatty monocarboxylic acid to a reactor and maintaining said fatty monocarboxylic acid at an elevated temperature while mixing;

(iii) adding said hot caustic solution to said fatty monocarboxylic acid in said reactor; and

(iv) either (a) feeding said acyl isethionate salt to said reactor at a time prior to the addition of said caustic solution and then adding said hot caustic solution at a temperature of at least 80°C in step (iii) or (b) feeding said acyl

isethionate salt to said reactor at a time subsequent to step (iii).

4. A process according to any one of the preceding claims which is for continuous production of a said composition, the process comprising the steps of:

(i) forming an aqueous caustic solution, at an elevated temperature, comprising sodium hydroxide and alkali metal isethionate;

(ii) separately and simultaneously charging a first feed stream of said  $C_8-C_{22}$  alkyl fatty monocarboxylic acid and a second feed stream of said hot caustic solution into a mixing chamber to form said alkali metal  $C_8-C_{22}$  alkyl fatty monocarboxylic acid salt; and

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(iii) mixing said formed alkali metal  $C_8-C_{22}$  alkyl fatty monocarboxylic acid salt with said alkali metal  $C_8-C_{22}$  acyl isethionate salt to form said composition, and wherein either said acyl isethionate salt is introduced to said mixing chamber as a slurry in said fatty monocarboxylic acid in which case said feed stream of said hot caustic solution is introduced into said mixing chamber at a temperature of at least  $80^{\circ}C$ , or said acyl isethionate salt is introduced subsequent to step (ii).

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Form 10

COMPLETE SPECIFICATION

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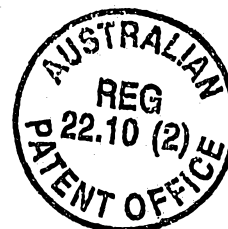
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TO BE COMPLETED BY APPLICANT

Name of Applicant:

UNILEVER PLC

Address of Applicant: UNILEVER HOUSE  
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Actual Inventor:

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Complete Specification for the invention entitled:  
PROCESS FOR PREPARING SOAP-ACYL ISETHIONATE  
COMPOSITIONS.

The following statement is a full description of this invention  
including the best method of performing it known to me:-

PROCESS FOR PREPARING SOAP-ACYL ISETHIONATE  
COMPOSITIONS

5           The invention relates to a process for  
          detergent  
preparing compositions comprising a major amount of  
soap and a minor amount of acyl isethionate.

10           Soap is an excellent cleaning agent but is  
          quite harsh to the skin. A study by Frosh & Kligman,  
          J. Amer. Acaderm. pp. 35 (1979), revealed that  
          substantial replacement of soap with an alternative  
          detergent such as an acyl fatty isethionate would  
          provide a more skin compatible system. Unfortunately,  
          this alternative is expensive. Less costly solutions  
          are needed to provide the consumer with an economical,  
15           yet mild, product.

          One approach to resolving the problem has  
          been reported in US Patent 4,695,395 (Caswell et al.).  
          The patent reports that bars containing a major amount  
          of soap and a minor amount of acyl isethionate can be  
20           rendered relatively non-irritating by incorporation of  
          non-acylated sodium isethionate. Consequent upon this  
          discovery, there was a need for a process to prepare  
          such compositions.

          US Patent 4,663,070 (Dobrovolny et al.)  
25           discloses a batch process wherein a reactor containing  
          a major amount of soap, a minor amount of  $C_{10}$ - $C_{16}$  acyl

isethionate, sodium isethionate, water, stearic acid, sodium chloride and certain minor additives are heated at 99°C-104°C under agitation. The reaction was judged as complete and terminated when the blend had passed a second peak in viscosity.

A related case, US Patent 4,707,288 (Irlam et al.) reports an essentially identical formulation prepared in a reactor under conditions of shear maintaining a temperature of from 60°C to about 90°C. Thereafter, the composition is fed to a plodder and extruded to form a detergent bar.

Each of the foregoing processes begin with soap as a starting material. A necessary condition for the soap/acyl isethionate based mixing is the need for certain initial levels of water. Without a minimum water level in the raw materials, blending would be difficult and a gritty product would result. A disadvantage of the aforementioned processes containing water is that moisture must be reduced through evaporation to arrive at an acceptable end product. There is a critical window of moisture beyond which bar physical properties are adversely affected. A second problem with the aforementioned processes is the time required in blending soap with acyl isethionate before there can be achieved the appropriate product viscosity.

Elimination of a drying step has long been known in the soap making art. For instance, US Patent 2,578,366 (Mills) meters an aqueous sodium hydroxide stream and a fatty acid slurry stream into a mixing reactor. Typically, the aqueous solution of caustic soda is maintained at about 27°C-35°C. Temperatures for the neutralization and subsequent soap mixing range from about 55°C to 102°C.

Along similar lines, US Patent 3,657,146 (Framson et al.) reveals a method for the direct production of soap from fatty acid under reaction temperatures of 120°C-180°C. Separate streams of tallow/coconut (80/20) fatty acids, of a stoichiometric amount of aqueous sodium hydroxide, and of sodium chloride are pumped into a reactor vessel.

In principle, it would appear attractive to form soap in situ, neutralizing fatty acid, while simultaneously feeding acyl isethionate into the blend. However, there is a problem. Acyl isethionate is susceptible to hydrolysis. This route would therefore not be perceived as feasible.

It is an object of the present invention to provide a process for preparing compositions containing a major amount of soap and a minor amount of acyl isethionate, and which are suitable for shaping into toilet bars.

Further objects of at least some forms of

this invention is to provide a method for producing a soap/acyl isethionate composition which is suitable for shaping into toilet bars and substantially eliminates the need for drying and thereby increases production rates, and/or provides a process route which  
5 minimizes hydrolysis of the acyl isethionate component, and/or yields a soap/acyl isethionate composition having consumer use and toilet bar processing properties that fall within commercially acceptable parameters.

10 The present invention provides a process for the production of a composition comprising alkali metal salts of  $C_8-C_{22}$  alkyl fatty monocarboxylic acid and  $C_8-C_{22}$  acyl isethionate in a weight ratio of 20:1 to 1:0.98, said process comprising the steps of:

15 (i) forming hot aqueous caustic solution comprising sodium hydroxide and alkali metal isethionate.

(ii) charging  $C_8-C_{22}$  alkyl fatty monocarboxylic acid to a reactor and maintaining said  
20 fatty monocarboxylic acid at an elevated temperature while mixing;

(iii) adding said hot caustic solution to said fatty monocarboxylic acid in said reactor; and

(iv) either (a) feeding said acyl  
25 isethionate salt to said reactor at a time prior to the addition of said caustic solution in step (iii) or

subsequent to step (iii) and adding said hot caustic solution at a temperature of at least 80°C in step (iii) or (b) feeding said acyl isethionate salt to said reactor at a time subsequent to step (iii).

5           The caustic solution is preferably added at a temperature of at least 80°C in any event. More preferably it is maintained and added at a temperature of 90°C-95°C, desirably at about 93°C. The amount of sodium hydroxide should of course be substantially  
10 stoichiometric, i.e. sufficient to bring about substantially complete neutralization of the fatty acid. The caustic solution will typically contain at least 30% by weight solids (sodium hydroxide and sodium isethionate).

15           The process may be carried out as a batch process, in which the hot caustic solution is added slowly to the fatty monocarboxylic acid in the reactor.

          There is also provided a process for continuous production of a composition comprising  
20 alkali metal salts of  $C_8$ - $C_{22}$  alkyl fatty acid and  $C_8$ - $C_{22}$  acyl isethionate in a weight ratio of about 20:1 to 1:0.98, said process comprising the steps of:

          (i)           forming a hot aqueous caustic solution comprising sodium hydroxide and alkali metal  
25 isethionate.

          (ii)          separately and simultaneously

charging a first feed stream of said  $C_8-C_{22}$  alkyl fatty monocarboxylic acid and a second feed stream of said hot caustic solution into a mixing chamber to form said alkali metal  $C_8-C_{22}$  alkyl fatty monocarboxylic acid salt; and

(iii) mixing said formed alkali metal  $C_8-C_{22}$  alkyl fatty monocarboxylic acid salt with said alkali metal  $C_8-C_{22}$  acyl isethionate salt to form said composition, and wherein either said acyl isethionate salt is introduced to said mixing chamber as a slurry in said fatty monocarboxylic acid in which case said feed stream of said hot caustic solution is introduced into said mixing chamber at a temperature of at least  $80^\circ\text{C}$ , or said acyl isethionate salt is introduced subsequent to step (ii).

Embodiments of the invention will now be described. Parts and percentages are by weight, unless the contrary is indicated. Reference will be made to the accompanying drawing which is a schematic flow diagram for a continuous process utilizing an extruder as reactor.

Both a batch and continuous method for the production of soap/acyl isethionate compositions will now be described. These methods utilize fatty acids as a starting material for the soap. Unless otherwise stated, parameters found for the batch route are

equally relevant to that of the continuous one.

Broadly, a batch process embodying one form of this invention involves mixing together acyl isethionate and distilled fatty acids to produce a slurry in a reactor vessel. Under agitation, the fatty acids in the slurry are neutralized by slow addition to the vessel of a hot caustic solution comprising sodium hydroxide, sodium isethionate and water. After a mixing period, e.g. about 30 minutes, the resultant blend can be discharged from the vessel for further processing including cooling on chill rolls, milling, plodding and stamping operations to form the composition into bars.

In this form of the invention, a factor of importance in obtaining a composition that minimizes the hydrolysis of the acyl isethionate concerns that of the caustic solution temperature. The temperature of this solution must be maintained at a temperature of at least 80°C, preferably about 93°C. Lower temperatures result in a substantial crystallization of caustic and increased acyl isethionate hydrolysis.

Other parameters can also have some effect upon acyl isethionate hydrolysis. Best yields are obtained where there is sufficient electrolyte present in the caustic solution to achieve a saturated state. Typical electrolytes are alkali metal and alkaline earth chloride and sulfate salts, especially sodium

chloride. Amounts of electrolyte that will normally achieve the saturation level will range from about 0.4% to about 2%, preferably from 0.4% to 1.5%, optimally about 0.8% by weight based on the weight of the final product composition.

The amount of alkali metal isethionate may be from 1% to 20%, preferably from 2% to 10% of the final product composition. Sodium isethionate is convenient.

It has also been found desirable that there be slow addition, accompanied by high rotational speed mixing, of caustic solution to the fatty acid/acyl isethionate components.

If the process is carried out as a continuous process, a feed of fatty acid is generally injected into a reactor upstream from where the caustic solution enters. An extruder is a suitable reactor vessel for this process. Fatty acids are normally held in their feed vessel at about 93°C and thus are fed in the molten state to the extruder. Acyl isethionate may be introduced in combination with the molten fatty acid. Alternatively, acyl isethionate may be introduced into the reactor at a point downstream from where the caustic solution enters. Further ingredients such as stearic acid should be dosed to the reactor at a point downstream from the aqueous caustic entry point.

EXAMPLE 1Batch Process

5 In a batch process, processing commenced with molten tallow fatty acid and cocoyl isethionate being charged into a Patterson mixer. These two ingredients were mixed and heated for no longer than 10 minutes. Mixing and heating produced an off-white suspension or slurry of cocoyl isethionate/tallow fatty acid.

10 Neutralization of the fatty acids was conducted at about 93°C. The Patterson mixer was heated with steam to this temperature. Upon achieving this temperature, a caustic solution of sodium hydroxide, sodium isethionate and water which had been  
15 preheated to 93°C, was metered by peristaltic pump into the Patterson mixer containing the cocoyl isethionate/tallow fatty acid batch. Addition was performed at a rate such that the caustic solution was completely added within 4 to 5 minutes. During the  
20 latter part of this time interval, normally after about 4.5 minutes, viscosity of the mixture increased to the point where it became semi-solid. Phase transition of material in the reactor was accompanied by a substantial energy release. A brief temperature rise  
25 of around 11°C occurred but temperature returned to the set point within 3-5 minutes.

After addition of the caustic solution, the batch was mixed at 93°C for at least 30 minutes. Stearic acid was then added and mixing continued for an additional 5 minutes. Thereafter, the product was removed from the reactor and chill rolled into ribbons. Perfume, preservatives and other minor ingredients were surface coated onto the ribbons. Further processing included conventional milling, plodding and stamping to obtain final bars. The finished, saponified formulation, ignoring hydrolysis of cocoyl isethionate, is outlined in Table I.

TABLE I

Theoretical Final Composition (before hydrolysis)

| 15 | <u>Ingredient</u>                                                 | <u>Weight %</u> |
|----|-------------------------------------------------------------------|-----------------|
|    | Sodium fatty monocarboxylic acid salt (82/18 tallow/coconut soap) | 51.17           |
| 20 | Sodium cocoyl isethionate                                         | 21.93           |
|    | Stearic/palmitic acid                                             | 6.19            |
|    | Coconut fatty acid                                                | 1.33            |
| 25 | Sodium isethionate                                                | 5.00            |
|    | Water                                                             | 10.50           |
| 30 | Fragrance                                                         | 1.50            |
|    | Titanium dioxide                                                  | 1.00            |
|    | Sodium chloride                                                   | 0.43            |
| 35 | Miscellaneous minor ingredients                                   | 0.22            |

EXAMPLE 2

The effects of temperature were evaluated in a series of experiments outlined in Table II. Process and composition were essentially identical to that described in Example 1, except for variations of the caustic solution temperature.

TABLE II  
Percent Cocoyl Isethionate Loss by Varying  
Caustic Solution Temperatures

|    | Experiment No. | Electrolyte (Sodium Chloride) (% Wt/Wt Product) | Caustic Solution Temperature (°C) | % Cocoyl Isethionate Active Loss |
|----|----------------|-------------------------------------------------|-----------------------------------|----------------------------------|
| 15 | 1              | 0                                               | 93                                | 5.4                              |
|    | 2              | 0                                               | 65                                | 24.1                             |
| 20 | 3              | 0.43                                            | 93                                | 3.8                              |
|    | 4              | 0.43                                            | 71                                | 15.6                             |
|    | 5              | 0.86                                            | 93                                | 2.8                              |
| 25 | 6              | 0.86                                            | 65                                | 12.2                             |

From the Table, it is seen that the caustic solution temperature radically affects the hydrolysis (loss) of cocoyl isethionate. Experiments 1, 3 and 5 exhibit active loss of 5.4% or less. By contrast, at 65°C-71°C the loss is more than tripled falling within the range 12.2 to 24.1%. See Experiments 2, 4 and 6.

EXAMPLE 3

Electrolyte level also has some effect upon limiting the amount of cocoyl isethionate lost through hydrolysis. Table III sets forth relationship of electrolyte concentration to that of % cocoyl isethionate loss.

TABLE III

% Cocoyl Isethionate Loss by Varying Electrolyte  
Concentration

|    | Experiment<br>No. | Initial<br>Batch<br>Moisture<br>(%) | Electrolyte<br>(Sodium<br>Chloride<br>(% Wt/Wt Product) | Caustic<br>Solution<br>Temperature | % Cocoyl<br>Isethionate<br>Active Loss <sup>*</sup> |
|----|-------------------|-------------------------------------|---------------------------------------------------------|------------------------------------|-----------------------------------------------------|
| 5  |                   |                                     |                                                         |                                    |                                                     |
| 10 | 7a                | 14                                  | 0                                                       | Ambient                            | 11.53                                               |
|    | 8a                | 14                                  | 0.43                                                    | Ambient                            | 10.74                                               |
|    | 9a                | 14                                  | 0.86                                                    | Ambient                            | 9.60                                                |
| 15 | 10a               | 14                                  | 0.97                                                    | Ambient                            | 9.65                                                |
|    | 11a               | 14                                  | 1.08                                                    | Ambient                            | 8.96                                                |
| 20 | 12a               | 14                                  | 1.3                                                     | Ambient                            | 8.98                                                |
|    | 7b                | 18                                  | 0                                                       | Ambient                            | 11.63                                               |
|    | 8b                | 18                                  | 0.43                                                    | Ambient                            | 10.61                                               |
| 25 | 9b                | 18                                  | 0.86                                                    | Ambient                            | 9.79                                                |
|    | 10b               | 18                                  | 0.97                                                    | Ambient                            | 9.01                                                |
| 30 | 11b               | 18                                  | 1.08                                                    | Ambient                            | 8.97                                                |
|    | 12b               | 18                                  | 1.3                                                     | Ambient                            | 9.29                                                |
|    | 13                | 18                                  | 0                                                       | Ambient                            | 11.78                                               |
| 35 | 14                | 18                                  | 0                                                       | 93°C                               | 8.82                                                |
|    | 15                | 18                                  | 1.3                                                     | 93°C                               | 3.86                                                |

40 \*Note: Experiments No. 7b-12b are the average of at least 2 runs.

From Table III, it is evident that cocoyl isethionate hydrolysis is slowed in the presence of

certain amounts of salt. Yield improvement occurs up to a level of about 1.3% by weight electrolyte. Beyond levels of 2% electrolyte, other physical properties become evident such as that of unacceptable  
 5 mush values. A combination of a 1.3% salt level with a caustic temperature of 93°C was particularly effective in experiment No. 15 where only 3.86% hydrolysis occurred.

10 TABLE IV

% Cocoyl Isethionate Loss by Varying the Type of  
 Electrolyte

| Experiment No. | Electrolyte        | Electrolyte Concentration (% Wt/Wt Product) | % Cocoyl Isethionate Active Loss |
|----------------|--------------------|---------------------------------------------|----------------------------------|
| 15 16          | Potassium chloride | 0.86                                        | 8.93                             |
| 20 17          | Calcium chloride   | 0.86                                        | 8.98                             |
| 18             | Lithium chloride   | 0.86                                        | 9.29                             |
| 25 19          | Calcium sulfate    | 0.86                                        | 8.98                             |

From Table IV, it is evident that the particular type of electrolyte is not critical. Any inorganic salt that readily  
 30 dissolves in the caustic solution will be acceptable.

EXAMPLE 4

The following experiment was run similar to that described in Example 1 except that the caustic solution  
5 was added to the batch reactor concurrently with cocoyl isethionate/tallow fatty acid. Table V summarizes the parameters of these experiments.

From Table V, it is evident that by the concurrent addition of caustic solution with the other reactants,  
10 the amount of cocoyl isethionate loss is quite significant, ranging from 22.8 to 33.2.

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TABLE V

Batch Process with Simultaneous Addition of All Reactants

| Experi-<br>ment<br>No. | Process-<br>ing Temp-<br>erature<br>(°C) | % Water in<br>Batch After<br>Neutrali-<br>zation<br>is Complete | Caustic Solution Composition |                       |                         | Final<br>%<br>Cocoyl<br>Isethi-<br>onate | %<br>Cocoyl<br>Isethi-<br>onate<br>Loss* |
|------------------------|------------------------------------------|-----------------------------------------------------------------|------------------------------|-----------------------|-------------------------|------------------------------------------|------------------------------------------|
|                        |                                          |                                                                 | %<br>Water                   | % Sodium<br>Hydroxide | % Sodium<br>Isethionate |                                          |                                          |
| 20                     | 82                                       | 14.2                                                            | 56.3                         | 39.8                  | 3.9                     | 17.75                                    | 22.8                                     |
| 21                     | 93                                       | 14.2                                                            | 56.3                         | 39.8                  | 3.9                     | 16.55                                    | 28.2                                     |
| 22                     | 82                                       | 18.0                                                            | 63.3                         | 31.6                  | 3.1                     | 16.45                                    | 26.4                                     |
| 23                     | 93                                       | 18.0                                                            | 65.3                         | 31.6                  | 3.1                     | 15.9                                     | 33.2                                     |

Note:

\*Average of two runs

EXAMPLE 5

A procedure similar to Example 1 was again used, although without close regulation of processing temperature. In a control experiment, addition to the reactor followed the same sequence as in Example 1. In further experiments the addition of acyl isethionate was delayed until after the addition of caustic solution and consequent neutralization of the fatty acid to soap.

This led to reduced isethionate loss, as shown by the following Table VI

TABLE VI

Batch Process Studying Effects of Cocoyl  
Isethionate Point of Addition

5

|    | <u>Experiment<br/>No.</u>                                                                                                                                                                          | <u>Isethionate<br/>Addition<br/>Method</u> | <u>% Cocoyl<br/>Isethionate<br/>Loss*</u> |
|----|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------|-------------------------------------------|
| 10 | 24                                                                                                                                                                                                 | Control                                    | 23.0                                      |
|    | 25                                                                                                                                                                                                 | Modified                                   | 9.9                                       |
|    | 26                                                                                                                                                                                                 | Reverse                                    | 3.7                                       |
| 15 | *Control = cocoyl isethionate/tallow fatty acid added prior to caustic addition.                                                                                                                   |                                            |                                           |
| 20 | Modified = tallow fatty acid followed by slight excess caustic addition (to neutralize the coco fatty acid impurity in cocoyl isethionate) and cocoyl isethionate added last after soap formation. |                                            |                                           |
| 25 | Reverse = tallow fatty acid followed by equivalent weight caustic addition and cocoyl isethionate added last after soap formation.                                                                 |                                            |                                           |

EXAMPLE 6

This Example illustrates the continuous process outlined schematically in the sole drawing.

- 5 The drawing shows liquid streams being fed to a twin screw extruder 10 driven by an electric motor 11. More particularly, the extruder was a Werner and Pfleiderer Corporation 32L/D twin screw extruder having a 40 mm screw flight diameter. The first feed stream which entered along line 12 was a slurry of cocoyl isethionate/tallow fatty acid. Downstream therefrom was introduced along line 14 a second feed stream. This was a hot caustic solution comprising sodium hydroxide, sodium isethionate and water. Screw configuration of the extruder was such that caustic and fatty acid streams were rapidly completely neutralized and continue mixing as they travel towards discharge from the extruder 10. Residence times within the extruder normally ranged from 3 to 5 minutes, depending upon product throughput, rotational speed and configuration of the extruder screw.

- 25 The cocoyl isethionate/tallow fatty acid feed stream was prepared using a Schold intensive mixer (not shown). This stream was prepared by first adding molten tallow fatty acid to the mixer with agitation. After reaching a temperature of 93°C, cocoyl

isethionate was added to the molten tallow fatty acid. Heating at 93°C with mixing was continued for 20 minutes whereafter the mixture was transferred to a waiting extruder feed tank 18 in which a temperature of approximately 93°C was maintained.

Caustic solution was prepared by mixing water, 50 weight % sodium hydroxide, and 56 weight % sodium isethionate together in a feed tank 20. It was found necessary that the water be added first to the tank to provide sufficient solvent for the two solutes (sodium hydroxide and sodium isethionate) to avoid precipitation of the sodium hydroxide. Temperature maintained in this tank was approximately 93°C.

The extruder had a barrel whose length was divided into five 5/1 length/diameter sections that were controlled with separate dual output, self tuning controllers. These controllers regulated electrical heating or closed loop cooling of the respective barrel sections. Feed rates to the extruder were controlled via a K-tron loss-in-weight liquid feed system. Each feed tank rested on top of a scale 22 which relayed tank weight information to a controller. Changes in tank weight with time were monitored by the controller. The controller then regulated the rotational speed of a gear pump 24 feeding the extruder. The system automatically compensated for a decreasing suction head

occurring as a result of the liquid height in the feed tank decreasing as feeding progressed. At an entrance to the extruder, each feed line was fitted with an injection nozzle allowing feed to enter into the extruder under a specified pressure. Respective lines 26 provide for recycle to the feed tanks.

Upon start up, the caustic line was first opened and then the cocoyl isethionate/tallow fatty acid feed line opened thereafter. Extruder screw speed was set at 400 to 500 rpm. Feed line injection pressures were adjusted to 350 kN/m<sup>2</sup>. Feed controllers were normally set to achieve a product throughput of 91 Kg per hour.

Stearic acid was maintained at a temperature of 65°C in third feed tank 28. This was metered into the extruder 10 along line 30. The product was discharged from the extruder in the form of noodles 34.

With the above-identified equipment, a series of continuous runs were conducted. Table VII lists details of these experiments. The continuous process achieved a cocoyl isethionate loss of only 8-10 weight %. These results are significant considering that in batch experiments where a total caustic charge is added in a single shot, cocoyl isethionate hydrolysis is approximately 20-30%, and sandy bars result. The neutralization temperatures were measured values of the

extruder barrel jacket temperature.

TABLE VII

5

Continuous Extruder Processing

|    | Neutrali-<br>zation<br>Moisture<br>(%) | Neutrali-<br>zation<br>Temperature<br>(°C) | Sample<br>No. | % Cocoyl<br>Isethionate<br>Loss |
|----|----------------------------------------|--------------------------------------------|---------------|---------------------------------|
| 10 | 16.36                                  | 80                                         | 1             | 3.55                            |
|    |                                        | 100                                        | 2             | 5.41                            |
|    |                                        | 120                                        | 3             | 7.74                            |
| 15 | 14.98                                  | 80                                         | 1             | 8.03                            |
|    |                                        | 100                                        | 2             | 8.80                            |
|    |                                        | 120                                        | 3             | 9.70                            |
| 20 | 14.98                                  | 80                                         | 1             | 9.86                            |
|    |                                        | 100                                        | 2             | 8.28                            |
|    |                                        | 120                                        | 3             | 8.59                            |
| 25 | 15.01                                  | 80                                         | 1             | 9.82                            |
|    |                                        | 100                                        | 2             | 7.25                            |
|    |                                        | 120                                        | 3             | 9.03                            |
| 30 | 18.00                                  | 80                                         | 1             | 9.87                            |
|    |                                        | 100                                        | 2             | 9.50                            |
|    |                                        | 120                                        | 3             | 10.22                           |

## THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A process for the production of a detergent composition suitable for shaping into toilet bars,  
5 comprising alkali metal salts of  $C_8-C_{22}$  alkyl fatty monocarboxylic acid and  $C_8-C_{22}$  acyl isethionate in a weight ratio of 20:1 to 1:0.98, said process comprising the steps of:
- 10 (i) forming an aqueous caustic solution, at an elevated temperature, comprising sodium hydroxide and alkali metal isethionate;
- (ii) charging  $C_8-C_{22}$  alkyl fatty monocarboxylic  
15 acid to a reactor and maintaining said fatty monocarboxylic acid at an elevated temperature while mixing;
- (iii) adding said hot caustic solution to said fatty monocarboxylic acid in said reactor; and  
20
- (iv) either (a) feeding said acyl isethionate salt to said reactor at a time prior to the addition of said caustic solution and then adding said hot caustic solution at a temperature of at least  $80^{\circ}C$  in step (iii) or  
25 (b) feeding said acyl

isethionate salt to said reactor at a time subsequent to step (iii).

2. A process according to claim 1 wherein the acyl isethionate salt is added to the reactor in admixture with said fatty monocarboxylic acid.

3. A process according to claim 1 wherein the aqueous caustic solution is added at a temperature of at least 80°C, and the acyl isethionate salt is added subsequent to step (iii).

4. A process according to any one of the preceding claims which is for continuous production of a said composition, the process comprising the steps of:

(i) forming an aqueous caustic solution, at an elevated temperature, comprising sodium hydroxide and alkali metal isethionate;

(ii) separately and simultaneously charging a first feed stream of said  $C_8-C_{22}$  alkyl fatty monocarboxylic acid and a second feed stream of said hot caustic solution into a mixing chamber to form said alkali metal  $C_8-C_{22}$  alkyl fatty monocarboxylic acid salt; and

(iii) mixing said formed alkali metal  $C_8-C_{22}$  alkyl fatty monocarboxylic acid salt with said alkali metal  $C_8-C_{22}$  acyl isethionate salt to form said composition, and wherein either said acyl isethionate salt is introduced to said mixing chamber as a slurry in said fatty monocarboxylic acid in which case said feed stream of said hot caustic solution is introduced into said mixing chamber at a temperature of at least  $80^{\circ}C$ , or said acyl isethionate salt is introduced subsequent to step (ii).

5. A process according to claim 4 wherein the acyl isethionate salt is included in the first feed stream in admixture with said fatty monocarboxylic acid.

6. A process according to claim 4 or claim 5 wherein said first feed stream is introduced at a point upstream from where said caustic solution enters said mixing chamber.

7. A process according to any one of claims 4 to 6 wherein said acyl isethionate salt is fed into said mixing chamber at a point downstream from the entry points of both said caustic solution and said

fatty monocarboxylic acid streams.

8. A process according to any one of claims 4 to 7 wherein said mixing chamber is an extruder.

9. A process according to any one of the preceding claims wherein the caustic solution is added at a temperature of 90-95°C.

10. A process according to any one of the preceding claims wherein the aqueous caustic solution contains from 0.4 to 2% by weight of an electrolyte.

11. A process according to claim 10 wherein the aqueous caustic solution contains from 0.4 to 1.5% by weight of an electrolyte.

12. A process according to any one of the preceding claims wherein the aqueous caustic solution contains sodium hydroxide and alkali metal isethionate in a total amount of at least 30% by weight of the solution, and in proportions such that addition of solution in an amount substantially stoichiometric to the fatty acid introduces alkali

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metal isethionate in an amount from 1% to 20% of the overall composition.

13. A process according to any one of the preceding claims further comprising forming the composition into bars.

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