(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 236 984**
A2

(12) # EUROPEAN PATENT APPLICATION

(21) Application number: **87103251.2**

(51) Int. Cl.4: **C11D 1/06** , C11D 17/00

(22) Date of filing: **06.03.87**

(30) Priority: **08.03.86 NL 8600607**

(43) Date of publication of application:
16.09.87 Bulletin 87/38

(84) Designated Contracting States:
AT BE CH DE ES FR GB IT LI NL SE

(71) Applicant: **STAMICARBON B.V.**
Mijnweg 1
NL-6167 AC Geleen(NL)

(72) Inventor: **Smid, Jacob Kornelis**
J.G. Heuthorststraat 61
NL-7009 CL Doetinchem(NL)

(74) Representative: **Hoogstraten, Willem Cornelis Roeland et al**
OCTROOIBUREAU DSM Postbus 9
NL-6160 MA Geleen(NL)

(54) **Syndet soap.**

(57) Syndet soap with one or more carbonamidopolyethercarboxylic acid derivatives as detergent components. The detergent component consists of one or more compounds with the formula R-CO-NH-$(C_2H_4O)_2$ CH_2COOM, where R represents an aliphatic hydrocarbon residue with 9-17 carbon atoms and M an alkali metal-, magnesiumatom, ammonium-or substituted ammonium group.

EP 0 236 984 A2

SYNDET SOAP

The invention relates to syndet soap based on one or more carbonamidopolyethercarboxylic acid derivates. 'Syndet' is the customary contraction of 'synthetic detergent' and syndet soap is understood to mean a soap in which the alkali metal salts of fatty acids customary of old have been replaced wholly or largely by a synthetic detergent.

Carbonamidopolyethyercarboxylic acid derivatives known from EP-A-0102118 are particularly well compatible with the human skin, because they not only have a low primary, but also a low secondary toxicity, i.e. that they can be utilized as a nutritive source for the skin bacteria.

According to the process described in EP-A-0102118 for preparing carbonamidopolyethercarboxylic acid, i.e. compounds having the general formula $R\text{-}CO\text{-}NH\text{-}(C_2H_4O)_n\text{-}CH_2COOM$, where R represents an aliphatic hydrocarbon residue with 5-21 carbon atoms, M represents alkali metal, ammonium or amino and n is a number from 2-10, a mixture with diverse values of n is obtained. For soap tablets or soap powders the consistency is mostly not satisfactory. Such product mixtures are often pasty so that the processing thereof into finished products by mixing them with customary components such as binders, fillers, colourants and perfumes and the like is a difficult procedure, and these compositions cannot or hardly be processed to form soap powders with satisfactory powder characteristics and/or soap tablets.

It has now been found that carbonamidopolyethercarboxylic acid derivatives having the formula $R\text{-}CO\text{-}NH\text{-}(C_2H_4O)_2\text{-}CH_2COOM$, where R represents an aliphatic hydrocarbon residue with 9-17 carbon atoms and M represents an alkali metal-, magnesium atom, ammonium-or substituted ammonium group, are very suitable as detergent components of solid syndet soaps.

The carbonamidoethercarboxylic acid derivatives in question can be prepared easily by converting a carboxylic acid (or carboxylic acid mixture) with diglycolamine, followed by carboxymethylation known from, for instance, EP-A-0102118. Subsequently, the acid can then be converted into a salt thereof.

Similar compounds having only one C_2H_4O group in the molecule are known from DE-A-2,644,498 and can be prepared in a similar manner by using monoglycolamine instead of diglycolamine. These compounds are surface active and are not disturbing when present also in the syndet soap in question. In the preparation it is possible, therefore, to use diglycolamine containing minor amounts of monoglycolamine.

It should further be noted that the carbonamidodiethercarboxylic acid salts according to the present invention are suitable also for other uses in which solid detergents with good compatibility with the skin are desirable, for instance in carpet shampoos, as additives for fatty acid soaps or as additives for synthetic soaps based on other known surface-active agents.

By varying the fatty acid residue, RCO, and the salt the melting points of the compounds can be conveniently regulated and thus optimum combinations of properties for toilet soap can be achieved. Thus the RCO group may be derived from a single fatty acid or from a mixture of fatty acids (for instance obtained from a natural oil) with, in the latter case, of course, a lowering of the melting point. Further, the potassium salt is found to have a higher melting point than the sodium salt, which is unlike the ordinary soaps. Amine salts tend to be more pasty, but may therefore, mixed with relatively high-melting salts, lead to a suitable consistency again. As, further, compounds of this kind show very little sensitivity towards calcium, a magnesium salt, too, and in principle even a calcium salt can well be used.

Some of the salts that can be used according to the invention were prepared by converting the relative acid with diglycolamine, resp. monoglycolamine and carboxyethylation. The melting points of a few salts were determined and are as follows:

RCO	number of EP-units	salt	melting point
Lauryl	2	Na	90
Lauryl	2	K	118
Lauryl	2	Mg	62
Lauryl	2	triethylamine	waxy
Lauryl	1	Na	162
coconut oil fatty acid	2	Na	78

The present compounds were screened for their comptability with the skin in comparison with a few other synthetic detergents. The experiments were made according to the so-called Duhring chamber test, which is described by P.J. Frosch and A.M. Kligman in Contact Dermatitis 5, 73-81 (1979) and in J. Am. Acad. Dermatol. 1, 35-41 (1979). This test has been developed in order to quantify low irritation potentials of detergents or of similar products destined for the skin.

0.05 ml of the example to be examined is applied to parts marked out on the inside of the lower arm. This is done by means of the so-called finn chambers, aluminium chambers with a diameter of 12 mm containing, for the liquid preparations, paper filters with a diameter of 11 mm. On the first day the samples are left to stay on the skin for 18 hours, upon which an interval of 6 hours is observed. On the second up to and including the fifth day the samples are left on the skin for 6 hours each day. The skin irritations are determined on the eighth day using the following scale:

Erythema	0	negative
	1+	very light, point-shaped or diffuse erythema
	2+	well recognizable, clear-cut erythema, moderate, homogeneous
	3+	medium-strong erythema
	4+	strong, fiery red erythema with oedema or epidermal effect (blisters, necroses)
Scale formation	0	negative
	1+	dry
	2+	fine scale formation
	3+	moderate scale formation
	4+	strong scale formation with large flakes
Chapping	0	negative
	1+	very superficial separation in the epidermis, fine cracks
	2+	single or more wide chaps
	3+	deep chaps with bleeding or sweating out, formation of blisters.

The examination was carried out on 14 women and men aged 18-35 with healthy skins. For the duration of the experiment the guinea pigs did not use detergent substances and creams.

By way of comparison water and sodiumlaurylsulphate, which is known to be highly irritating to the skin, were examined, the latter as an 0.25% solution. Also examined were:

A: $C_{11}H_{23}CO\text{-}NH\text{-}(CH_2CH_2O)_2CH_2COON_a$ as a 10% solution in water;

B: a commercially available syndet soap as a 10% solution in water;

C: a syndet soap based on compound A, again as a 10% solution in water.

According to a brochure of the manufacturer, the commercial product used for the purpose of comparison contains the following components:

Combination of fatty alcohol (C_{12}-C_{18}) sulphate and disodium fatty alcohol (C_{12}-C_{18})-sulphosuccinate circa 50.0%

Filler (on polysaccharide basis) circa 23.0%

Plasticizer (waxy) circa 23.0%

Water 3.5 ± 1.5%

Titaniumdioxide circa 0.1%

The syndet soap C was composed as follows:
Compound A 25%
Lauridit V 254 (commercial product, mixture of fatty acid monoethanolamide, glycolmonostearate and glycoldistearate) 13.5%
Aminol KDE (commercial product, diethanolamide of coconut oil fatty acid as foam stabilizer) 4.5%
Polyethyleneglycol 12.000 20.0%
Letocil GAT (commercial product, glycolmonostearate) 5.0%
Hard paraffin 4.0%
Na_2SO_4 10.0%
Maize starch 15.0%
Titaniumdioxide 1.0%
Aluminiumtriformate 1.0%
Zincstearate 1.0%

The results, expressed as averages, are as follows:

	Erythema	Scaling	Chapping
Water	0	0	0
Sodiumlaurylsulphate (0.25%)	2.5	2.4	0.4
Compound A	0	0.4	0
B	3	3.4	1.3
C	0	0.4	0

As shown above, the sodiumlaurylsulphate-containing commercially available syndet soap is relatively highly irritating to the skin, whereas the present soap and the capillary active product used therein cause hardly any irritation of the skin.

The syndet soap (product C) according to the invention contains the hardness required for soap and foamed well.

Hardness and foaming of product C were compared with tablets of soap that had been made according to identical recipes, but with replacement of the detergent compound A by: D the corresponding com pound with only one ethoxy group and E: a compound similar to A in which, however, the lauryl residue has been replaced by the coconut oil fatty acid residue, so in fact by a mixture of acid residues.

It has been found that product D, as was to be expected, foams less well than product C and is harder, too. Product E has been found to foam even a little better than product C and is, as was to be expected, a little softer. It is clear that through suitable combination optimization is possible.

Claims

1. Syndet soap with one or more carbonamidopolyethercarboxylic acid derivatives as detergent components, characterized in that the detergent component substantially consists of one or more compounds with the formula $R\text{-}CO\text{-}NH\text{-}C_2H_4O)_2\text{-}CH_2COOM$, where R represents an aliphatic hydrocarbon residue with 9-17 carbon atoms and M an alkali metal-, magnesium atom, ammonium-or substituted ammonium group.

2. Syndet soap according to claim 1, characterized in that R represents the group $C_{11}H_{23}$.

3. Syndet soap according to claim 2, characterized in that M represents sodium.