PHILIPPINE PATENT [19]

[11] No.: 26543
[45] Issued: AUG 19 1992

[54] Title: SOAP COMPOSITION

[75] Inventor (s): MR. JOHN GEORGE CHAMBERS, MR. NORMAN HALL, MR. GEOFFREY IRLAM, all of Merseyside; DR. CAROLINE SUSAN CORDERY and MR. MICHAEL HOOD, both of Cheshire, all of England

[73] Assignee (s): UNILEVER NV, of Rotterdam Netherlands, a company of Netherlands

[22] Filed: September 29, 1989

[21] Application Serial No: 39311

FOREIGN APPLICATION PRIORITY DATA

[31] Number (s) : 8823656.7

[32] Date (s) : October 7, 1988

[33] Country (ies) : United Kingdom

[52] PH Class 252/135, 252/109

[51] Int. Class C11D 9/14

[58] Field of Search 252/135, 252/109

[56] Reference (s) Cited and/or Considered:

Chemical Abstract, Vol.95 p 2222208h

[57]

ABSTRACT see attached sheet

BAD ORIGINAL

26543

ABSTRACT OF THE DISCLOSURE

SOAP COMPOSITIONS

Toilet soap bar comprises 40 to 89 wt% soap (reckoned as anhydrous), 1 to 33 wt% tetrasodiumpyrophosphate (reckoned as anyhydrous $Na_4P_2O_7$), and (x + 19) % by weight water, where x% is the calculated amount of water required to effect complete hydration of the pyrophosphate to its decahydrate. The soap bar has a smaller content of soap than conventional soap bars and yet can retain acceptable yield stress.

SOAP COMPOSITIONS

The present invention relates to toilet soap compositions, particularly soap compositions in the form of toilet bars.

Toilet soap compositions in bar form typically contain 85 to 92% by weight of soaps and 8 to 15% by weight of water. The soap is usually derived from blends of oils and/or fatty acids with a blend iodine value of not more than 60, and the possibilities for incorporating filler materials are limited.

There are a number of constraints which compel the adoption of such a composition. Notably, a greater water content or the presence of more unsaturation, indicated by a higher iodine value, leads to a composition which is soft and sticky and cannot be processed into bars except with difficulty. Conventional extrusion equipment cannot handle such soft and sticky compositions while running at normal speeds. Increasing the water content of soap bars has the additional consequence that the bars tend to shrink on storage, leading to stress cracking.

Use of soluble fillers e.g. polyols tends to produce deterioration in properties perceived by the user, such as lather formation, bar hardness, rate of wear and development of surface mush.

To summarize, a successful toilet bar composition needs ideally to be capable of processing on conventional extrusion equipment (so the composition must not be too soft and sticky), must provide satisfactory lather and other user-perceived properties and must avoid cracking. A further pitfall which a soap composition must avoid is efflorescence, that is to say crystalline growth appearing on the surface of bars during processing, storage or use.

There have been a number of proposals to incorporate inorganic electrolytes into soap bars in varying amounts and for various reasons. See for example GB 1244346 (Colgate).

We have now found that the incorporation of one particular electrolyte makes it possible to avoid the constraints referred to above, while still maintaining satisfactory properties. By contrast we have found that this is not possible with other electrolytes we have tested.

According to a first aspect of the present invention there is provided a toilet soap bar comprising:

40 to 89% by weight soap (reckoned as anhydrous)

1 to 33% by weight tetrasodiumpyrophosphate (reckoned as anhydrous $Na_4P_2O_7$, and

(x + 4)% to (x +19) % by weight water, where x% is the calculated amount of water required to effect complete hydration of the pyrophosphate to its decahydrate.

Hydration of soduim pyrophosphate to its decahydrate proceeds by the reaction

$$Na_4P_2O_7 + 10\ H_2O \longrightarrow Na_4P_2O_7 . 10H_2O$$

The value if x can readily be calculated from the amount of pyrophosphate. The molecular weight of anhydrous $Na_4P_2O_7$ is 266. The molecular weight of water is 18. So, if the percentage by weight of anhydrous pyrophosphate is y %, the percentage of water is given by:

$$x\% = \frac{y\% \times 18 \times 10}{266} = 0.68y\%$$

In other words the amount of water is given by

$$(0.68y + 4)\ \% \leq x\% \leq (0.68y + 19\%)$$

where x% is the total amount of water by weight and y% is the amount of sodium pyrophosphate reckoned as anhydrous.

By including sodium pyrophosphate we have found that it is possible to lower the amount of soap in the composition to lie in the range from 40 to 89% by weight (reckoned as anhydrous) with a water content lying between 4.68 and 41.44%, while still retaining acceptable yield stress (i.e hardness) and also keeping other properties satisfactory. The amount of soap (reckoned as anhydrous) in the composition need be not more than 85%, or even not more than 80% by weight (reckoned as anhydrous soap). The level of sodium pyrophosphate preferably lies in a range from 10 to 30%

by weight. The actual proportion of ingredients selected in any one case will depend among other things on the desired properties of the finished bar and on for example the type of soap blend selected for inclusion in the bar. We have found for instance that less water is needed with soft oil blends than with hard oil blends (ie. IV < 60) for which a minimum total water content is preferably 9.68 wt%.

The hardness of a soap bar can be quantified by measuring the yield stress of the bar. Toilet soap bars embodying the present invention have a yield stress at 20°C of at least 3.0×10^5 Nm^{-2}, up to a preferred maximum yield stress value at 20°C of 10×10^5 Nm^{-2}. Measurement of yield stress is described inter alia in Elementary Rheology by G W Scott Blair, Academic Press, London 1969 and Rheometry: Industrial Applications Ed. K. Walters Research Studies Press (a division of John Wiley & Sons Ltd.) New York 1980.

Soap sources suitable for use in the present composition include conventional toilet soap sources such as tallow, hydrogenated tallow, hydrogenated rice bran oil, hydrogenated linseed oil, palm oil, hydrogenated palm oil, coconut oil and babassu oil in the usual soap blend proportions of 60 to 95 wt% tallow, hydrogenated tallow, hydrogenated rice bran oil, hydrogenated linseed oil, palm oil, hydrogenated palm oil, the like and mixtures thereof and 40 to 5 wt% coconut oil, babassu oil, the like and mixtures thereof. We have however found that it is possible to use in compositions embodying the present invention oil blends whose fatty acid moieties have a degree of olefinic unsaturation that is higher than usually used for a toilet soap bar. In particular we have found that the present composition permits the use of soap blends having an iodine value of more than 60. Examples of oils that can be employed containing a high degree of olefinic unsaturation include soyabean oil, sunflower oil, linseed oil, cottonseed oil and rice bran oil. The proportion of such oils present can be up to 100 wt% of the soap blend and can for instance be determined by the prevailing price of locally available materials. The soap bar could thus be made from a soap blend having an iodine value of up to 130. All the soaps present are preferably sodium soaps.

The present compositions permit the inclusion more water soluble filler than would be possible in the absence of sodium pyrophosphate. Examples of such water soluble fillers include sugars, glycerol, sorbitol and sucrose. The amount that it is possible to include will vary from filler to filler. In the case of sucrose it is possible to include between 0 and 10 wt% with respect to the final weight of the bar, without adversely effecting bar properties to an unacceptable degree.

It is possible to render the present compositions superfatted ie. to ensure the presence of free fatty acids in the bar either by adding the desired free fatty acids per se to the soap blend or by adding an acid such as citric acid or phosphoric acid to the soap blend which displaces free fatty acid from the soap mixture. Suitably the compositions can be superfatted so as to ensure the presence of 5 to 10 wt% free fatty acid with respect to the soap blend employed.

There are other salts beside sodium pyrophosphate which will take up water as water of crystallization. We have found however that none of these salts we have otherwise tested is suitable. In some cases the temperature at which the water of hydration is lost is relatively low, so as to be below the ambient temperature in hot climates. Additionally with all conventional hydratable salts except sodium pyrophosphate, we have observed efflorescence. Although we do not wish to be bound by any theory it would appear that the water solubility of an electrolyte determines whether efflorescence will occur from compositions which have amounts of soap and water in accordance with the present composition. There appears to be a rather low limit on water solubility, above which efflorescence is observed. Only tetrasodium pyrophosphate comes below this limit and hence provides a soap bar avoiding problems of efflorescence.

Compositions embodying the invention can be produced by mixing sodium pyrophosphate with moisture-containing soap. A preferred technique is to add anhydrous sodium pyrophosphate to so-called neat soap containing approximately 70% soap and 30% water with the neat soap in a molten state. The sodium pyrophosphate can be added as a dry powder, as a hot concentrated solution or as a slurry with a small amount of hot water. The soap mixture containing sodium pyrophosphate can then be dried to its intended water content, optionally by use of a vacuum flash dryer as is conventionally used for drying neat soap to the water content appropriate for conventional toilet soap. However, less drying will be required for the present compositions. An alternative processing route would be to meter the sodium pyrophosphate into the flow of neat soap as it goes to the dryer.

Another production route would be to mix the sodium pyrophosphate with soap which has been obtained by drying neat soap, e.g. soap noodles coming from a vacuum flash dryer. Mixing with such soap noodles can be carried out with a mixer which applies shear to the mixture.

By adding the sodium pyrophosphate at a relatively early stage in the processing of the soap into bars, the sodium pyrophosphate introduced into the composition reaches its hydrated state at an early stage, giving a composition which can be subjected to conventional subsequent processing steps. Normally such steps includes admixing of minor ingredients such as dyes, perfumes, antioxidants, preservatives, e.g. with a chip mixer, milling, plodding, extruding and stamping into bars.

In a second aspect therefore the present invention provides a process for preparing a soap composition according to the first aspect of the present invention, comprising admixing tetrasodiumpyrophosphate at a level of 1 to 33 wt% calculated with respect to the final toilet soap bar composition with moisture containing soap.

Suitably the moisture containing soap has a water content in excess of 20 wt% and the soap is subjected to a drying step. Conveniently the moisture containing soap is neat soap.

Generally the resulting composition will then be milled, plodded and shaped into bars.

The present invention will now be described further and exemplified by the following Examples in which all percentages are by weight.

Example 1

A number of bars were prepared with varying contents of soap, water and tetrasodium pyrophosphate.

The bars were prepared on a pilot plant, adding tetrasodium pyrophosphate in the form of a slurry to neat soap (approximately 30 % wt water content) thereafter drying to the desired water content, milling, plodding and stamping into bars. The pyrophosphate slurries were freshly made using an amount of water calculated to be 6% by weight of the final composition. The neat soap was a conventional 80/20 tallow/coconut soap.

The yield stress of the bars at 20°C was determined by observation of the extent to which a bar was cut by a weighted cheese wire during a specified time. A horizontally braced cheese wire of diameter d cm suspended from a counterbalanced and freely pivotable arm was brought into contact with a freshly prepared bar of soap cooled to room temperature (20°C). A corner edge of the soap was positioned under the wire such that when a weight W gm was placed on the arm directly above the cheese wire, the length of cut L cm made by the wire increases to the limit where the stress exerted by the wire equals the resistance of the bar. The stress exerted by the wire at this limit is equal to the yield stress of the soap bar. The time taken to reach this limit was of the order of 30 seconds. In practice a standard 1 minute cut time was allowed in each case.

For a bar having an orthogonal corner edge in tranverse cross section the yield stress of the bar was calculated using the following formula:

$$\text{Yield stress} = \frac{3}{8} \times \frac{W98.1}{L\ d}\ Nm^{-2}$$

The in-use properties of the bars, specifically rate of wear, mush formation during use and lather generation, were tested using bars which had been stored at room temperature of 20°C for at least four weeks and examined for appearance.

Bars were stored under accelerated ageing conditions and examined at intervals. The storage conditions comprised storing for one week at 20°C with 90% relative humidity then for one week at 37°C with 70% relative humidity and repeating this cycle for several months while examining the appearance of the bars each week.

The various compositions and their yield stress values are set out in the following Table 1 where the term "free water (theory)" signifies the amount of water calculated to remain after full hydration of the pyrophosphate. In the table the control composition is a conventional 80/20 tallow/coconut soap bar composition without sodium pyrophosphate.

TABLE 1

SAMPLE	% SOAP (anhy.)	% $Na_4P_2O_7$ (anhy.)	% WATER	% FREE WATER (theory)	YIELD STRESS at 20°C (Nm^{-2})
Control	88	0	12	12	4.0×10^5
A1	78	4	18	15.3	3.0×10^5
A	60	15	25	14.8	3.9×10^5
B	56	15	29	18.8	3.5×10^5
C	55	15	30	19.8	2.5×10^5
D	53	15	32	21.8	1.2×10^5
E	56	20	24	10.5	7.5×10^5
F	50	20	30	16.5	3.1×10^5
G	45	20	35	21.5	1.0×10^5
H	40	20	40	26.5	0.6×10^5
I	45	36	19	-5.4	21.8×10^5
J	45	15	40	29.8	0.2×10^5
K	44	26	30	12.4	6.1×10^5
L	33	33	34	11.6	4.8×10^5

The compositions in Table 1 embodying the present invention were all observed to have a yield stress greater than 3.0×10^5 n.m.$^{-2}$, which is the minimum hardness for a composition capable of processing into toilet bars using conventional equipment.

By contrast the comparative compositions C, D, G, H and J with a theoretical free water content greater than that permitted by the present invention had insufficient hardness.

Composition I had insufficient water to achieve a full hydration of the pyrophosphate, giving a negative theoretical value for free water. The resulting composition was found to be extremely hard and its in-use properties were poor, that is to say it had a low rate of wear and low lather formation. It was also observed to give efflorescence during storage. Composition L had insufficient soap to meet the requirements of the present invention. Its hardness was satisfactory but its in-use properties were poor. The in-use properties of the compositions which did lie within the requirements of this invention were in every case found to be satisfactory.

Measurements of the rate of wear, mush and lather characteristics of bars control, A1, A, F, K and L were made and the results are given in Table 2 below.

TABLE 2

SAMPLE	RATE OF WEAR (%)	IN USE MUSH	LATHER (mls)
Control	23	6	41
A1	28	13	46
A	21	6.5	35
F	22	12	35
K	17	4	34
L	16	5	29

The results in Table 2 show that bars embodying the present invention have comparable properties to the control bar having a higher soap content and containing no sodium pyrophosphate.

Example 2

A number of bars were made in the same manner as for Example 1 but in place of tetrasodium pyrophosphate other hydratable salts were employed. The bars were stored under accelerated ageing conditions and appearance of the bars was noted at intervals. The compositions and the results of the storage tests are set out in Table 3 below: as noted in the table all the bars gave an unsatisfactory appearance with efflorescence apparent at some stage.

Table 3

ELECTROLYTE	% SOAP (anhy.)	% ELECTROL. (anhy.)	FREE WATER (theory)	RESULT
Sodium sulphate	78	3.9	13.2	E,C,PE
Sodium carbonate	78	3.9	11.5	E,C
Sodium metaborate	78	3.9	13.8	IE,E
Trisodium orthophosphate	59.2	12.0	13.0	E
Disodium hydrogen orthophosphate	61.8	10.4	12.0	E

E = efflorescence, IE = efflorescence in use,

C = cracking, PE = efflorescence during processing

Example 3

Bars were made in the same manner as for Example 1, with varying amounts of sucrose incorporated with the tetrasodium pyrophosphate. Yield stress of the bars was measured at 20°C. In-use properties were observed, using bars which had been stored at 20°C for at least four weeks. The in-use property of mush formation was measured by means of a test consisting of weighing the amount of mush scraped from a bar which has been allowed to stand in a specified depth of water for a specified time.

The compositions and results are set out in Table 4.

TABLE 4

SAMPLE	SOAP wt% (anhy.)	$Na_4P_2O_7$ wt%	WATER wt%	SUCROSE wt%	YIELD STRESS (Nm^{-2})	IN-USE MUSH
Contrl1	86	0	14.0	0	4.0×10^5	9.0
Cntrl2	70	0	30.0	0	1.6×10^5	17.0
M	63	0	27.0	10	2.2×10^5	30.0
N	54.5	15	25.5	5	3.9×10^5	12.4
P	50.5	15	24.5	10	2.8×10^5	17.0

In the table the first control is a conventional 80/20 tallow/coconut toilet soap composition. The second control is neat soap, which had much lower hardness and higher mush development. Comparative composition M shows that addition of sucrose without tetrasodium pyrophospate gives an inadequate improvement in hardness and excessive mush formation. Composition N which includes tetrasodium pyrophosphate has satisfactory hardness and much less mush formation. Composition P with a greater level of sucrose and with tetrasodium pyrophosphate has satisfactory hardness.

Example 4

A number of bars were made in the same manner as for Example 1, but employing blends of oils for the soap base which have an iodine value of more than 60. Two different oil blends were used. In one (Q) the mixture was a blend of 32 wt% tallow, 48 wt% soyabean oil and 20 wt% coconut oil having an overall iodine value of 83. In the other blend (R) a mixture of 80 wt % soyabean oil and 20 wt% coconut oil was employed having an overall iodine value of 110. The compositional make-up of the bars and the yield stress value measured at 20°C for each type of bar is given in Table 5 below.

Table 5

SAMPLE	SOAP (wt%)	SODIUM PYROPHOSPHATE (wt%)	WATER (wt%)	YIELD STRESS (Nm^{-2})
Q	58	20	22	3.4×10^5
R	54	25	21	3.9×10^5

In each case the bars had a yield stress of more than 3×10^5 Nm^{-2} and thus provided toilet bars of acceptable hardness. Comparative bars containing soaps from the same oil blends but with no added tetrasodium pyrophosphate were extremely soft and sticky and impossible to process into bars on conventional extrusion equipment. Bar Q had the following in-use properties: rate of wear 17%, in use mush 6 and lather volume 36 mls, which characteristics are comparable to those of the control bar employed in Example 1.

Example 5

A number of bars were made in the same manner as for Example 1. The present bars were made however from a 60/40 blend of tallow and coconut soaps which was superfatted ie. contained free fatty acids (FFA). The amount of free fatty acid present is expressed as a percentage of the tallow/coconut soap blend present ie. a 60/40, 5 blend means a blend of soaps comprising 60 wt% tallow soap and 40 wt% coconut oil soap in which is additionally present 5 wt% free fatty acid with respect to the soap blend.

The formulation details are given in Table 6 below.

Table 6

SAMPLE	SOAP/FFA CHARGE	SOAP/FFA (wt%)	SODIUM PYROPHOSPHATE (wt%) (anhydrous)	WATER (wt%)
S	60/40, 5	61	15	24
T	60/40, 10	66	15	19
Control	60/40, 7	88	0	12

For each type of bar its yield stress at 20°C, rate of wear, mush and lather characteristics were assessed. The results are given in Table 7 below.

Table 7

EXAMPLE	YIELD STRESS (Nm^{-2})	WEAR (%)	OBJECTIVE MUSH	LATHER VOLUME
S	3.1×10^5	20	7	47
T	3.1×10^5	18	5	66
Control	3.0×10^5	17	10	53

The results in Table 7 show that superfatted bars containing tetrasodium pyrophosphate have in use and hardness properties comparable to superfatted bars containing no pyrophosphate and hence correspondingly more of the soap/fat charge.

CLAIMS

1. Toilet soap bar comprising:

40 to 89% by weight soap (reckoned as anhydrous)

1 to 33% by weight tetrasodiumpyrophosphate (reckoned as anhydrous $Na_4P_2O_7$), and (x + 4) % to (x + 19) % by weight water, where x% is the calculated amount of water required to effect complete hydration of the pyrosphosphate to its decahydrate, and is equivalent to 0.68 of the percentage by weight of tetrasodiumpyrophosphate computed as 0.68 of 1 to 33% thus giving a total water content in the bar lying between 4.68 wt% and 41.44%.

2. Toilet soap bar according to claim 1 wherein the soap content (reckoned as anhydrous) is from 40 to 85 wt%.

3. Toilet soap bar according to claim 2 wherein the soap content (reckoned as anhydrous) is from 40 to 80 wt%.

4. Toilet soap bar according to claim 1 wherein the level of tetrasodiumpyrophosphate (reckoned as anhydrous) lies in a range from 10 to 30% by weight with respect to the final toilet soap bar composition.

5. Toilet soap bar according to claim 1 wherein the soap comprises a blend of soaps whose fatty acid

moieties have an iodine value in the range from 60 to 130.

6. Toilet soap bar according to claim 1 wherein the bar contains free fatty acids within the range 5 to 10 wt% with respect to the soap blend employed.

7. Toilet soap bar according to claim 1 wherein the bar contains a water soluble filler.

8. Toilet soap bar according to claim 1 having a yield stress at 20°C lying within the range 3.0×10^5 Nm^{-2} to 10×10^5 Nm^{-2}.

JOHN GEORGE CHAMBERS
CAROLINE SUSAN CORDERY
NORMAN HALL
MICHAEL HOOD
GEOFFREY IRLAM
Inventors