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ANTI-BACTERIAL DETERGENT BAR

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ABSTRACT OF THE DISCLOSURE

An improved anti-bacterial detergent bar is obtained by incorporating an anti-bacterial compound in the mixture from which the bar is formed in a potentiator. Anti-bacterial compounds to which the improvement can be applied have a minimum inhibitory concentration against Staph. aureus of less than 5 p.p.m., a solubility in water of less than 1,000 p.p.m. at 20° C. and a melting point of at least 70° C. The potentiator is a solvent capable of dissolving at 25° C. at least 25% of its own weight of the anti-bacterial compound.

This is a continuation of application Ser. No. 744,686, filed July 15, 1968, and now abandoned.

This invention relates to an anti-bacterial detergent bar.

The final stages in the production of a bar usually include a step in which a mixture based on a detergent is worked, usually under conditions of shear. The mixture is then shaped to form the bar, usually by plodding and stamping. Materials which can be present in the final bar in small amounts include perfume, opacifiers, whitening agents, colourants, antioxidants and sequestering agents. When an anti-bacterial bar is to be made, the anti-bacterial compound is usually added with the other additives immediately prior to or during the step in which the mixture is worked. The anti-bacterial compounds normally employed in the manufacture of anti-bacterial bars are solid organic compounds. For an anti-bacterial compound to be suitable for use in a bar it must be compatible with the detergent concerned and have an MIC value (minimum inhibitory concentration) against Staph. aureus of no more than 5 p.p.m. Preferably the anti-bacterial compound should have an MIC value against Staph. aureus of less than 2 p.p.m. and particularly preferably less than 1 p.p.m.

It has now been discovered that the anti-bacterial activity of a bar containing an anti-bacterial compound having certain characteristics defined herein can be enhanced by adding the anti-bacterial compound to the mixture from which the bar is produced in a potentiator as herein defined.

The enhancement can be assessed by measurement of the reduction on the hands of flora which are normally present on the skin. These flora are believed to act on sebaceous and other secretions of the skin, particularly the secretion of the apoorine sweat gland, to produce typical body odour.

The invention is applicable to anti-bacterial compounds that substantially do not dissolve or melt during the working in the final stages of the preparation of the bar. Although in general the solubility of a substance in a detergent bar does not correlate very well with its solubility in water it has been found that for solid organic anti-bacterial compounds there is a rough correlation between their solubility in most detergent bars and their solubility in water. In order to avoid undue experimentation it is recommended that it be assumed that if an anti-bacterial compound has a solubility in water at 20° C. of no more than 1,000 p.p.m. it is unlikely to dissolve in a detergent bar and as such the present invention is applicable to the anti-bacterial compound. It will be appreciated that there are exceptions to the above recommendation. These will normally be easily recognized. The maximum temperature normally attained in the final stages of the preparation of a bar is about 70° C. The invention is therefore applicable to such anti-bacterial compounds that have melting points of at least 70° C.; or more particularly to those that have melting points of at least 100° C.

A potentiator for an anti-bacterial compound is herein defined to be a solvent capable of dissolving at 25° C. at least 25% of its own weight of the anti-bacterial compound. A preferred potentiator is a solvent capable of dissolving at 25° C. at least 60% of its own weight of the anti-bacterial compound.

Although potentiators are preferred that are liquids or low-melting solids, substances can be used as solvents for the anti-bacterial compound that do not melt or soften until temperatures above 25° C. When such substances are used, they are heated with the anti-bacterial compound, for example on a steam bath, until solution is attained and then the mixture is cooled to 25° C. to give a solid solution. The mixture can be examined under a microscope to see whether the anti-bacterial compound has remained in solution: if it has the solvent is to be considered as a potentiator as herein defined.

A feature of the invention is the provision of a solution of a solid, organic anti-bacterial compound in a potentiator as herein defined.

It will be appreciated that, before an anti-bacterial compound is included in a bar, its biological properties and its effect on the properties of the bar will have been studied. Before any potentiator is included in the bar its effect on the properties of the bar and its biological properties will have been studied. The maximum amount of any potentiator that can be included will, like the amount of any particular antibacterial compound, vary from compound to compound. In general it is preferred to include no more than 10% by weight of the potentiator; even more preferably no more than 5% by weight of the potentiator should be included. The properties of the bar that the potentiator can adversely affect include the firmness of the bar, mush formation, lathering properties, colour stability and cracking.

The amount of total anti-bacterial compound in the bar should, in general, be no more than 4% by weight of the bar and preferably no more than 2%. Amounts of anti-bacterial compound as low as 0.05% by weight of the bar are effective.

Preferably the detergent on which the bar is based is soap. The nature of the soap base used to prepare the soap is not important. The soap will preferably be an alkali-metal, water-soluble soap of higher fatty acids with 10 to 18 carbon atoms. The alkali-metal should preferably be sodium. The soap bar will normally contain up to 15% by weight of water, preferably from 5 to 12% and minor amounts of electroylte such as sodium chloride, usually no more than 1%.

Soap bars according to the invention can, for example, be prepared from fat charges having the following composition (parts by weight):

Tallow fat	Coconut/palm kernel oil	Sperm oil distillate	Fatty acids (super fat)
70	20	10	
30	70		10
50	50		10
80	20		

The invention is particularly effective in anti-bacterial bars containing a minor proportion of superfatting agent. A typical superfatting agent consists of a mixture of higher fatty acids with 10 to 18 carbon atoms; a typical superfatted anti-bacterial bar normally contains between 1 to 20% of such a mixture.

The invention is applicable to an anti-bacterial soap bar containing an anti-bacterial compound and a non-soap detergent compound or mixture of non-soap detergent compounds as well as to an anti-bacterial non-soap detergent bar.

Among the classes of non-soap detergents which can be used in the manufacture of an anti-bacterial bar according to the invention are alkane sulphonates, alkene sulphonates, alkylbenzene sulphonates, alcohol sulphates, alcohol ethoxylate sulphates, monoglyceride sulphates, alkanolamide sulphosuccinates, alkyl sulpho-acetates, carboxylic acid alpha-sulphonates, acyl isothionates, acyltaurides, acyl-N-methyl taurides and acylamino methane sulphonates. Particular examples of alcohol sulphates are C_8 to C_{22} aliphatic alcohol sulphates containing 75% straight-chain and 25% 1-methyl alkyl groups.

The non-soap detergents are normally present at least mainly as their sodium salts although the invention is also applicable to anti-bacterial bars in which the non-soap detergent is present in the form of other alkali-metal, alkaline-earth, ammonium or organic base salts.

Particular examples of non-soap detergent active compounds which can be used in compositions of non-soap detergent toilet bars are those described in U.S. applications S.N. 618,333, 621,134, 621,150, all of which are now abandoned, 611,206 now Patent No. 3,523,089, and 705,022, now abandoned.

Although the mode of action of a potentiator is not fully understood it is believed that the potentiator affects the manner of flocculation and subsequent deposition on the skin of the anti-bacterial compound during washing. It is noticeable that during the personal washing operation although the surface of an anti-bacterial soap bar containing a potentiator remains free, or almost free, of anti-bacterial particles, often a high density of small (e.g. 0.5 to 1 micron or less in dimeter) particles is present in the wash lather. In some cases it is difficult to detect the particles in the lather because of their size or their optical properties. For instance particles of TCC (3,4,4'-trichlorocarbanilide) are more readily detected than those of TBS (3,5,4'-tribromosalicylanilide).

A slightly different mode of action is thought to apply when the potentiator is a substantially hydrophobic solvent. In this case, the solvent may act as a vehicle by which the anti-bacterial compound is deposited on the skin. The hydrophobic solvent is in some cases deposited during the personal washing operation on the skin as fine droplets.

For the potentiator to act successfully it is essential that the amount of potentiator in the bar should be greater than the amount of the potentiator capable of dissolving at 25° C. the amount of anti-bacterial compound it is intended to potentiate.

The required amount of the potentiator will vary depending upon the nature of the potentiator. In general there will be no advantage gained by adding more than 8 times by weight the amount of the potentiator capable of dissolving at 25° C. the amount of anti-bacterial compound it is intended to potentiate. When more than 8 times by weight is used there will in some cases be a reduction in the potentiation achieved and there will be a tendency for other properties of the bar, as mentioned above, to be affected adversely. It is preferred to use less than 4 times by weight the amount of the potentiator capable of dissolving at 25° C. the amount of anti-bacterial compound it is intended to potentiate.

Advantageously, particularly when high levels of the potentiator affect the properties of the bar adversely, only a portion of the anti-bacterial compound in the bar is potentiated. For example, if a bar contains 0.5% of TCC and 0.5% TBS sufficient potentiator can be included in the bar to potentiate just half the amount of total anti-bacterial compounds present. In such a case the bar will contain normal discrete particles of anti-bacterial compound but, in comparison with the bar containing no potentiator, the amount of these particles present will be about half.

When partial potentiation is not involved a particular aspect of the invention is the provision of an anti-bacterial soap, containing an anti-bacterial compound as herein defined in which no particles of the anti-bacterial compound can be detected optically. The invention can provide a transparent anti-bacterial bar; not attainable before with the comparatively insoluble anti-bacterial compounds to which the invention applies.

The amount of the potentiator in the bar, although necessarily greater than the amount required to dissolve at 25° C. the portion of anti-bacterial compound it is required to potentiate, need not be as great when the potentiator is a substantially hydrophobic solvent as when it is a more hydrophilic solvent.

In general when the potentiator is substantially hydrophobic the amount of potentiator in the bar should be from 1.2 to 4 times by weight of the amount of the potentiator capable of dissolving at 25° C. the portion of anti-bacterial compound it is intended to potentiate.

When the potentiator is a solvent with both hydrophilic and hydrophobic properties the amount of the potentiator in the bar should be 2 to 4 times by weight the amount of the potentiator capable of dissolving at 25° C. the portion of anti-bacterial compound it is intended to potentiate.

It is preferred to dissolve the amount of anti-bacterial compound it is intended to potentiate in the potentiator before incorporation of both in the mixture based on a detergent from which mixture the bar is formed by working and shaping.

It is also preferred to ensure that, after the anti-bacterial compound and potentiator have been added to the detergent mixture, the mixture is not worked too hard. In particular it is preferred if the temperature of the detergent after the anti-bacterial compound and potentiator have been added is not allowed to rise above 50° C., particularly preferably not above 35° C.

It is also preferred if the anti-bacterial compound and the potentiator are added at a late stage in the processing of the detergent mixture. Preferably the anti-bacterial compound and the potentiator should be added to the mixture after the mixture has been milled at least once after other additives, such as preservatives and dyestuffs, have been added. Such additives are often added in aqueous solution and it is preferred to avoid contracting the anti-bacterial compound and potentiator unnecessarily with water.

For the above reasons it is also preferred to incorporate the anti-bacterial and potentiator in the detergent substantially at the expense of water that is normally present. This means that the mixture into which the anti-bacterial compound and the potentiator are incorporated is dried more than normally.

Examples of anti-bacterial compounds to which the invention is applicable and that have been used and strongly recommended for use in detergent bars are given in Table I.

TABLE I

Anti-bacterial compound	Melting point, ° C.	Solubility at 25° C. in— Water	Soap	Minimum inhibitory concentration v. *Staph. aureus*, p.p.m.
3,4,4'-trichlorocarbanilide	252	Virtually insoluble	Substantially insoluble	0.1-0.2
3-trifluoromethyl-4,4'-dichlorocarbanilide	211-212	do	do	0.2-0.4
3,5,4'-tribromosalicylanilide	224-228	Sparingly soluble	do	0.8-1.0
3,5,3',4'-tetrachlorosalicylanilide	160-162	do	do	0.1-0.2
3,5,-dibromo-2'-trifluoromethylsalicylanilide		do	do	0.1-0.2
2,2'-dihydroxy-3,3',5,5',6,6'-hexachlorodiphenylmethane	161	do	Soluble	0.3-0.6
2,2'-dihydroxy-3,3',5,5'-tetrachlorodiphenylmethane	166	do	do	
2,2'-dihydroxy-3,3',dibromo-5,5'-dichlorodiphenylmethane	187-188	do	do	
2,2'-dihydroxy-3,3',5,5'-tetrachloro-diphenylsulphide	187-188	do	do	
2-hydroxy-4,4'-dichlorodiphenylether	78-79	do	do	0.1
2-hydroxy-4,2',4'-trichlorodiphenylether	56	do	do	0.5-2.0
2-hydroxy-3,5,4'-tribromodiphenylether	110-112	do	do	1.5-3.0
Tetramethylthiuramdisulphide	149-151	Insoluble	Substantially insoluble	0.2-2.0
2-mercaptopyridine-N-oxide (Zn complex)	265	do	do	

Potentiators that are particularly effective include: alkylphosphoramides, such as hexamethylphosphoramide; alkylureas, such as tetramethylurea; mixtures of C_8 to C_{22} fatty acids and polyethylene glycols; anionic-cationic complexes formed by mixing, if necessary with warming, amines and acids, preferably organic. The amines can be aliphatic primary, secondary or tertiary amines or aliphatic or alkylaryl quaternary ammonium compounds. The amines should normally include a long-chain fatty group and the total number of carbon atoms in the amine should preferably be at least 10. Preferred acids are straight-chain carboxylic acids. A preferred carboxylic acid is an alkylethoxyacetic acid having the general formula

$$R(OCH_2CH_2)_nOCH_2CO_2H,$$

where n is 1 to 25, preferably 1 to 10, and R is a long-chain C_8 to C_{22} group. The total number of carbon atoms in the cationic and anionic parts of the complex preferably should be at least 10 and less than 40, and even more preferably 10 to 30; phosphine oxides such as trioctylphosphine oxide; amine oxides such as lauryldimethylamine-N-oxide; and complexes formed from amine oxides and carboxylic acids such as acetic acid; substituted ammonium phosphates such as the salt formed between triethanolamine-6-EO and ortho- or pyro-phosphoric acid.

Table II gives a list of potentiators together with the solubilities of TCC and TBS in these potentiators.

TABLE II

	Percent solubility at 25° C. of N TCC	TBS
Polyethyleneglycols		
polyethyleneglycol 600 (mol wt.)	30	45
Polyethyleneglycol 1,000 (mol wt.)	35	45
Polyethyleneglycol mono-esters: Polyethyleneglycol 400 (mol wt.) monolaurate	35	40
Glycerolalkoxylates:		
glycerol 12 (mole) ethoxylate	30	30
glycerol 18 (mole) ethoxylate	35	35
Tetra-alkylureas: Tetramethylurea	50	45
Hexa-alkylphosphoramides: Hexamethylphosphoramide	70	40
Polyalkylpolyphosphoramides: Octamethylpyrophosphoramide	25	60
Amine oxides: Lauryldimethylamine-N-oxide	45	110
Amine oxide carboxylates:		
Lauryldimethylamine-N-oxide acetate	50	95
Lauryldimethylamine-N-oxide palm kernel carboxylate	25	45
Amine oxide sulphonates: Lauryldimethylamine dodecylbenzenesulphonate	35	25
Phosphine oxides:		
Tri-n-octylphosphine oxide	55	100
Triethanolamine		120
Triethanolamine alkoxylates: Triethanolamine 6 ethoxylate	35	60
Triethanolamine ethoxylate complexes: Triethanolamine-6-ethoxylate acetate	40	95
Triethanolamine-6-ethoxylate plam kernel complexes:		
Triethanolamine-6-ethoxylate lactate	45	55
Triethanolamine-6-ethoxylate succinate	35	80
Triethanolamine-6-ethoxylate citrate	45	65
Triethanolamine-6-ethoxylate benzoate	35	75
Triethanolamine-6-ethoxylate phthalate	45	65
Triethanolamine-6-ethoxylate terephthalate	55	35
Triethanolamine ethoxylate sulphonates: Triethanolamine-6-ethoxylate dodecylbenzene sulphonate	45	25
Mono-alkylamine complexes: Octylamine palm kernel carboxylate	35	

TABLE II.—Continued

	Percent solubility at 25° C. of— TCC	TBS
Mono-alkylamine ethoxylate complexes:		
Stearyl amine-10-ethoxylate lactate	55	75
Stearyl amine-10-ethoxylate succinate	45	75
Stearyl amine-10-ethoxylate citrate	55	70
Stearyl amine-10-ethoxylate benzoate	45	85
Stearyl amine-10-ethoxylate phthalate	45	55
Stearyl amine-10-ethoxylate terephthalate	45	35
di-Alkylamine complexes: Dicoconutamine palm kernel carboxylate	55	
Tri-alkylamine complexes:		
Cetyldimethylamine acetate	45	35
Cetyldimethylamine palm kernel carboxylate	40	
Cetyldimethylamine stearate	25	
Cetyldimethylamine oleate	25	85
Cetyldimethylamine glycolate	50	80
Cetyldimethylamine lactate	55	65
Cetyldimethylamine succinate	55	55
Cetyldimethylamine citrate	65	55
Cetyldimethylamine benzoate	55	85
Cetyldimethylamine phthalate	65	75
Cetyldimethylamine terephthalate	75	75
Tri-alkylamine sulphonates: Cetyldimethylamine dodecylbenzene sulphonate	45	35
Polyalkylenepolyamines: Polyethyleneimine 600	110	110
Polyalkylenepolyamine alkoxylates:		
Diethylenetriamine-5-propoxylate	175	175
Diethylenetriamine-20-propoxylate	175	175
Quaternary ammonium alkane complexes:		
Cetyltrimethylammonium soap*	45	25
Cetylbenzyldimethylammonium soap*	40	35

*Soap base: 80 tallow, 15 coconut fat, 5 sperm oil distillate.

Table III lists a number of suitable anionic moieties for anionic-cationic complexes.

TABLE III

Acetate
Alkylacetate
Hydroxyalkylacetate
Alkoxyethoxyacetate
Hydroxyalkoxyethoxyacetate
Amidoacetate
N-alkylamidoacetate
Amidoalkylacetate
Mono-alkene carboxylate
N-alkylamidoalkylacetate
Di-alkane carboxylate
Amidoalkoxyacetate
N-alkylamidoethoxyacetate
Alkyl, hydroxyalkyl, alkoxy can be C_{2-20}
Phosphate
Sulphate

Sulphonate

Alkylsulphonate
Hydroxyalkylsulphonate
Alkenylsulphonate
Hydroxyalkenylsulphonate
Arylsulphonate
Alkylarylsulphonate
Alkylethanolamidosulphosuccinate
Alkyl, hydroxyalkyl, alkenyl, hydroxyalkenyl can be C_{2-20}

The potentiating effect can be measured by means of Finger Imprint Tests and Handwashing Tests described

below. The Handwashing Test procedure is time-consuming and the procedure is also awkward in that, when either the anti-bacterial compound or the potentiator has not been sufficiently tested, they cannot be used even in a handwashing test without preliminary biological clearance. It has been found by experiment that the results of Finger Imprint Tests can be used to assess the probable behaviour of a bar in Handwashing tests. It is therefore possible to screen a number of possible potentiators. It will be appreciated that before a potentiator/anti-bacterial compound system can be recommended for use, biological clearance must be obtained and Handwashing Tests made.

washing of the hands with soap solutions containing anti-bacterial, subjects washed the hands with test soap bars containing anti-bacterial with and without potentiator. Also, the scale of evaluation of the high level of anti-bacterial or anti-bacterial mixture was effectively expanded by use of a larger volume of media and inoculum than that described by Vinson, and in addition plates were prepared as described by Vinson, but in which *E. coli* was substituted for *Staph. aureus.*

Table IV gives results for Finger Imprint Tests in soap bars formed from a fat charge comprising 80 parts by weight tallow, 15 coconut oil and 5 sperm oil distillate.

TABLE IV

Soap bar	TBS	TCC	TFC*	Potentiator	Percent	Finger imprint v. *Staph. a.*	Finger imprint v. *E. coli*
A				PEG_{400} monolaurate**	3	0	0
B		2				1.2	0
C	2					2.5	0
D		1.5					
		0.5		PEG_{1000}	4	1.8	0
E	1.5						
	0.5			PEG_{1000}	5	3.5	1.2
F	0.5	1.5				1.7	0
G	1.5	0.5				2.5	0
H		1.5					
	0.5			PEG_{1000}	5	3.8	1.7
I	1.5						
		0.5		PEG_{400} monolaurate	4	3.7	1.6
J		1.5	0.5			1.4	0
K		1.0	0.5	PEG_{400} monolaurate	4	3.1	0.5
		0.5					
L		1.0	0.5			1.5	0
M		1.0					
			0.5	Cetyldimethylammonium acetate	5	2.7	1.1

*3-trifluoromethyl-4,4'-dichlorocarbanilide.
**PEG_{400} monolaurate is the monolaurate of a polyethyleneglycol with an average molecular weight of 400.

Finger Imprint Test

The Finger Imprint Test used was a modification of the of L. J. Vinson et al., J. Pharm. Sci., 1961, *50*, (10), 827, "In Vitro Tests for Measuring Anti-bacterial Activity of Toilet Soap and Detergent Bars:" instead of simulated

One surprising and significant effect is the effect of potentiated anti-bacterial compounds on *E. coli*, a gram-negative organism.

Tables V and VI give the results from two further series of tests using smaller volumes of media and inoculum.

TABLE V

Soap bar	Potentiator (2%)	TCC	TBS	TFC	Finge imprin v. *Staph aureu*
A		1			0.8
B			1		2.2
C				1	1.6
D	Hexadecyldimethylamine phthalate	0.5			1.6
E	do		0.5		1.5
F	do			0.5	1.5
G	Triethanolamine ethyleneoxide lactate	0.5			2.0
H	do		0.5		2.0
I	do			0.5	3.2
J	Triethanolamine ethyleneoxide phthalate	0.5			1.7
K	do		0.5		2.5
L	do			0.5	2.2
M	Triethanolamine ethyleneoxide phosphate	0.5			1.2
N	do		0.5		1.7
O	do			0.5	2.7
P	Stearylamine 10 ethyleneoxide lactate	0.5			1.6
Q			0.5		1.7
R				0.5	2.9
S	Stearyl 10-ethyleneoxide sulphate	0.5			1.2
T	Stearylamine 10-ethyleneoxide mulphate		0.5		2.5
U	do			0.5	2.8
V	Hexamethylphosphoramide	0.5			2.7
W	do		0.5		2.4
X	do			0.5	2.5
Y	Tri-n-octyl phosphine oxide	0.5			1.2
Z	do		0.5		3.6
AA	do			0.5	1.7
AB	Dimethylformamide	0.5			1.3
AC	Diethylenetriamine 5 propyleneoxide	0.5			1.4

TABLE VI

Soap bar	Potentiator (2%)	TCC	TBS	TFC	*Finger imprint v. Staph. aureus*
A		1			0.6
B			1		3.0
C				1	1.7
D	Dimethylformamide		0.5		3.7
E	do			0.5	2.9
F	Dimethylenetriamine 5 propyleneoxide		0.5		3.3
G	do			0.5	2.8
H	Methyl fatty imidazoline ether propionate/secondary C_{11-15} alcohol 12 ethyleneoxide.		0.5		3.6
I	do			0.5	2.5
J	Dicoconutamine palm kernel acid carboxylate		0.5		3.6
K	do			0.5	2.4
L	Hexadecyldimethylamine laurylethoxyacetic acid carboxylate.	0.5			1.6
M	Hexadecyldimethylamine laurylethoxyacetic acid carboxylate.		0.5		3.6
N	do			0.5	2.7
O	Hexadecyldimethylamine/triethanolamine ethyleneoxide/succinic acid carboxylate.	0.5			0.9
Q	do		0.5		2.2
R	do			0.5	2.8
S	Lauryldimethylamine oxide	0.5			1.1
T	do		0.5		1.8
U	do			0.5	2.7
V	Lauryldimethylamine oxide/acetate	0.5			1.1
W	do		0.5		1.8
X	do			0.5	1.4
Y	Benzyltriethanolammonium ethyleneoxide chloride.	0.5			0.9
Z	do		0.5		2.4
AA	do			0.5	1.9

Handwashing Test

The method used has been described by B. M. Gibbs and L. W. Stuttard, J. Appl. Bact., *30*, (1), 1967, "Evaluation of Anti-bacterial Soap."

A panel of subjects were given control (free of anti-bacterial agent) soap for regular use, one week before the test began. They were then given either a control soap or a soap containing an anti-bacterial compound with or without potentiator, according to a statistically randomised design. During the test period, the subjects used only the prescribed soaps both at work and at home, and washed their hands with the prescribed soaps at least 3 times a day. On the sixth and seventh days, the resident flora of their hands were evaluated in the laboratory by the method described below. The soaps were then collected and the second week's soaps distributed, according to the statistical arrangement (when soaps contained 1.0% anti-bacterial, the test weeks were separated by a week in which subjects used only control soap—this prevented residual effects). The bacteria on the hands of the subjects were again evaluated on the sixth and seventh days, and the third week's soap issued, etc. The test continued in this way for 4 weeks.

For the laboratory evaluations, each subject made 4 consecutive washes with control soap, the first three up to the elbows, whereas in the fourth, the hands and wrists only were included. Fifteen seconds' soaping, 60 seconds' lathering and 15 seconds' rinsing were used in each case. This fourth wash was done in a bowl containing 2 litres of sterile water. The bacteria in each millilitre of water were counted on yeastrel-glucose agar using the poured plate technique and diluting the samples in sterile 0.1% peptone water when necessary (usually 1:10 gave a countable number of colonies). After incubation at 37° C. for 48 hours the number of colonies was counted and the count per bowl determined. For each subject, the mean of the counts on the two days was taken as subject's count for the week. This is considered to be proportional to the number of bacteria constituting the flora.

TABLE VII

Soap bar	TCC, percent	TBS, percent	Potentiator, percent		Reduction in skin flora, log counts per 0.1 ml. wash liquor
1	1.0				1.24736
2	0.5		Coconut monoethanolamide 4-5 ethoxylate	7.5	1.13236
3	0.5		Mono-n-butyl di-2-hydroxyethylether	7.5	1.15216
4	1.0				1.20986
5	0.5		Polyethyleneglycol 1,000 (mol. wt.)	4.0	1.05729
6	0.5		Polyethyleneglycol 400 monolaurate	4.0	1.10747
7	0.5		Coconut monoethanolamide 14 ethoxylate	6.0	1.27722
8	1.0				1.49969
9	0.75		Polyethyleneglycol 400 monolaurate	6.0	1.23848
10	0.75		do	8.0	1.29388
11	0.75		do	10.0	1.23268
12	1.5		do	8.0	1.08721
13	1.5		do	10.0	0.96149
14	0.5		do		1.84005
15	1.0				1.50941
16	0.5		Coconut diethanolamide 25 ethoxylate	5.0	1.58245
17	0.5		do	10.0	1.44355
18	0.5		Polyethyleneglyool 1,000	8.0	1.61012
19	2.0				1.07302
20		2.0			0.92572
21	1.5 0.5		Polyethyleneglycol 1,000	4	0.82802
22		1.5 0.5	do	5	0.79938
23	1.5	0.5			0.62581
24	0.5	1.5			0.70966
25	1.5	0.5	Polyethyleneglycol 1,000	5	0.55560
26	0.5	1.5	do	4	0.50927

TABLE VII—Continued

Soap bar	TCC, percent	Potentiator, percent		Reduction in skin flora, log counts per 0.1 ml. wash liquor
27	0.5			1.45410
28	1.0			1.23306
29	0.5	Coconut diethanolamide 25 ethoxylate	4	1.22588
30	0.5	Polyethyleneglycol 1,000 (mol wt.)	5	1.18959
31	1.0			1.27627
32	1.0	Polyethyleneglycol 400 monolaurate	7.4	0.93134

What is claimed is:

1. In a process for preparing an anti-bacterial soap bar in which process from 0.05 percent to 4 percent, by weight of the bar, of a saop compatible anti-bacterial compound, having a minimum inhibitory concentration against *Staph. aureus* of less than 5 p.p.m., a solubility in water of less than 1,000 p.p.m. at 25° C. and a melting point of at least 70° C. and selected from the group consisting of 3,4,4'-trichlorocarbanilide, 3-trifluoromethyl-4,4' - dichlorocarbanilide, 3,5,4' - tribromosalicylanilide, 3,5,3',4' - tetrachlorosalicylancilide, 3,5, - dibromo-2'-trifluoromethylsalicylanilide and combinations thereof, is added to soap and the soap is worked and shaped to form the soap bar; the improvement, by which the anti-bacterial effect is enhanced, being that the anti-bacterial compound is added to the soap with a solvent having hydrophobic and hydrophilic properties compatible with the bar and capable of dissolving at 35° C. at least 25 percent of its own weight of the anti-bacterial compounds, the amount of the solvent in the bar being by weight from 2 to 4 times the minimum amount of the solvent required to dissolve the anti-bacterial compound and being more than 0.05 percent and no more than 10 percent by weight of the bar.

2. A process according to Claim 1 in which a mixture of C_{10} to C_{18} fatty acids is added to form from 1 percent to 20 percent by weight of the bar.

3. A soap bar prepared in accordance with the process of Claim 1.

References Cited

UNITED STATES PATENTS

3,043,778	7/1962	Kelly	252—107
3,152,039	10/1964	Mattson	252—107 X
3,179,596	4/1965	Farrar et al.	252—125
3,226,329	12/1965	MacMillan	252—107
3,431,207	3/1969	Russell	252—106
3,485,919	12/1969	Markgraf et al.	424—322
3,594,322	7/1971	Wilson	252—106

FOREIGN PATENTS

1,013,040	8/1957	Germany.

OTHER REFERENCES

"TCC," Monsanto Technical Bulletin No. FC–4A, pp. 1, 6, 11, 19 and 20.

Linfield et al., "Performance of Germicidal and Deodorant Soaps," JAOCS, vol. 37, May 1960, pp. 251–254.

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P. F. WILLIS, Assistant Examiner

U.S. Cl. X.R.

252—106

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,835,057 Dated September 10, 1974

Inventor(s) Wai Ming Cheng, James Francis Davies and Brian Anthony Pethica

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 11, line 29 (Claim 1), change "35°C." to --25°C.--

Signed and sealed this 18th day of February 1975.

(SEAL)
Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents
and Trademarks

FORM PO-1050 (10-69)

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