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ABSTRACT

The invention relates to non-soap laundry bars suitable for hand washing of fabrics. Conventionally such bars are constituted by a dispersion of solid particles dispersed in a continuous phase which consists principally of detergent active and water. The bars of this invention have a continuous phase formed principally by alkali metal silicate and water with other materials either dispersed or dissolved in it. This structure provides bars having hardness, wear-rate and mashing characteristics which are very satisfactory.

SPECIFICATION

TO THE DIRECTOR OF PATENTS:

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DETERGENT COMPOSITIONS

This invention relates to detergent compositions constituting non-soap detergent NSD laundry bars. The use of such bars is in the hand washing of fabrics.

Laundry bars for the handwashing of fabrics, where the bars are applied directly to the fabric, must have structural integrity which is retained during handling after manufacture, transport and use. They must have adequate hardness and an appropriate rate of wear during use.

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Such bars typically contain an anionic detergent active together with various solids which provide the functions of detergency builder and filler, and also contribute to the structure of the bar. A quantity of moisture is normally present and the bars are normally prepared by a process in which the constituents of the bar are mixed together to form a dough which is subjected to considerable mechanical shear, after which the dough is shaped into bars.

Many published bar compositions include substantial quantities of sodium tripolyphosphate which functions as a builder and also contributes to the structure of the bar. It is also well known to incorporate alkali metal silicate into bars. A number of documents including GB 1230483, US 4150001

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and US/3178370 disclose bar compositions containing silicate with phosphate present in substantial quantities. At least some of these documents also describe compositions containing silicate with phosphate absent.

5 Incorporation of substantial levels of alkali metal silicate has been found to be somewhat undesirable in normal bar compositions, because it gives the bars an unpleasant feel during use.

 The object of the present invention is to provide
10 NSD laundry bars which have satisfactory properties and yet where the materials in the bar other than the detergent active itself can be of relatively low cost.

 Conventional NSD bars are a dispersion of solid particles in a continuous phase which consists principally of
15 detergent active and water. This can readily be demonstrated by submerging such bars in toluene for 4 days. This organic solvent dissolves the detergent active. Consequently it removes the continuous phase from the bars which therefore disintegrate or become fragile and crumbling after the
20 immersion in toluene.

 We have found that by appropriate choice of the levels of silicate and detergent active it is possible to produce bar compositions which have a continuous phase formed principally by alkali metal/silicate and water rather than
25 formed principally by the detergent active. Other materials will be dispersed in this silicate-based continuous phase,

and there may be some material dissolved in it also.

Accordingly, therefore, in a first aspect of the present invention we provide a shaped laundry bar of a composition including non-soap detergent active and alkali metal silicate, wherein the amounts of detergent active and silicate are such that the bar has an inorganic, silicate-containing continuous phase.

Preferably the bar will contain inorganic filler and an inorganic electrolyte which is a hydratable salt or the hydrated form of such a salt.

We have found that such laundry bars can have satisfactory properties and yet be formed from ingredients giving a relatively low overall cost in relation to the amount of detergent active provided within the composition.

The presence of a silicate-containing continuous phase is detectable by immersion of the bars in toluene at 20°C for 4 days. Conventional bars with a detergent active continuous phase lose their structural integrity under such conditions. Those with a silicate-containing continuous phase do not. Therefore in a second aspect this invention provides a shaped laundry bar of a composition including non-soap detergent active and alkali metal silicate wherein the amounts of detergent active and silicate are such that the bar retains structural integrity on immersion in toluene for 4 days at 20°C.

The silicate-containing continuous phase provides

coherence, structural integrity and a measure of flexibility which avoids brittleness. By itself, it can lead to bars which are rather too soft and sticky. It is preferred to incorporate into the bar composition a hydratable inorganic salt capable of taking up some water as water of crystallization. This leads to a harder bar - although the hardness depends also on the nature of the continuous phase. Because the salt hydrates it can be expected to be in its hydrated form, or partially so, in the bars.

It is desirable to use a highly absorbent salt, preferably tri alkali metal orthophosphate or (less preferred) penta alkali metal tripolyphosphate with a high phase I content. However, the amount of such phosphate should desirably not be too high, since it leads to unnecessary hardness. Too much orthophosphate can also lead to a gritty feel. A satisfactory level of phosphate will generally be not greater than 10 or 12% by weight of the composition, preferably not over 7% or even 5% of the composition. This approximates to a phosphorus content generally not greater than 2-2.5% and preferably not over 1.5 or even 1% by weight of the composition.

Orthophosphate appears to be more effective as a hardening agent when the continuous phase is silicate-based than when a detergent-based continuous phase is present.

A possible alternative to phosphate as a hydratable salt is alkali metal carbonate, bicarbonate or

sesquicarbonate.

In a third aspect, therefore, this invention provides a shaped laundry bar of a composition including non-soap detergent active, alkali metal silicate, and hydratable
5 inorganic electrolyte, possibly in the hydrated form thereof, wherein the amount of alkali metal silicate is at least 5% by weight of the composition (calculated on anhydrous silicate), and the amount of phosphorus, as inorganic phosphate, in the composition is not over 5% by weight of the composition. The
10 amount of phosphate may be at least 0.5% of the composition (i.e. at least 0.1% phosphorus).

The silicate employed in bars of this invention may be alkaline silicate, metasilicate or neutral silicate. We have found alkaline silicate, with a 2:1/ratio of SiO_2 : Na_2O
15 to be suitable.

The amount of silicate in bars of this invention will generally be between 5 and 20% by weight of the composition. Greater amounts can be used, but do not appear to confer any benefit.

20 Our UK Published Patent Application 2099013 describes a process in which aluminium silicate is generated in situ while mixing bar ingredients. Such aluminium silicate, formed in situ, is a useful bar-structuring material and may be employed in conjunction with this
25 invention.

The amount of detergent active in bars with a

silicate based continuous phase can be varied over a range.

This in itself is an advantage, in enabling formulations of bars so as to meet market requirements. The amount of detergent active will generally not exceed 35% by weight of the composition, and is also limited by the amount of silicate present. Too much detergent active leads to a continuous phase based on detergent active. It is likely that the amount of detergent active will not exceed 25% by weight of the composition.

The maximum amount of detergent active which can be included while retaining a silicate-based continuous phase can be determined experimentally, by immersing trial bars in toluene for 4 days and observing the state of the bars thereafter.

It is believed that, at least when alkyl benzene sulphonate is employed as the detergent active, a practical upper limit on the quantity of this detergent active is given by the formula:

$$x = 1/2 y + 14$$

where x% is the upper limit amount of detergent active (as a percentage by weight of the composition) and y% is the weight percentage of alkali metal silicate in the composition.

The amount of detergent active is preferably at least 10% by weight of the composition. A lesser amount may be employed but we prefer not to do so since we regard this as an excessive reduction in the quality of the bar as a

laundrying product. In a composition containing 10 to 35 active detergent, the ratio of detergent active to silicate may be approximately 2:1 by weight, e.g. between 1.75:1 and 2.25:1.

5 The detergent-active components usable in the present invention are well characterised in detergent bar technology. These components are characterized in "Surface Active Agents" Volume 1, by Schwartz and Perry (Interscience, 1958). Suitable detergent actives are found in the general
10 class of anionic actives, and can be used in admixture with nonionic, amphoteric, betaine and zwitterionic actives.

A preferred detergent-active component comprises linear or branched-chain alkyl benzene sulphonates (having from 8 to 16 carbon atoms in the alkyl chain), either alone
15 or in admixture with other actives, preferably in minor amounts to the alkyl benzene sulphonate. Specific examples of detergent actives usable as alternatives or in admixture with alkyl benzene sulphonates are alkane sulphonates, alcohol sulphates, especially C_{10} to C_{14} alcohol sulphates,
20 olefin sulphonates, monocarboxylic acid salts, ethoxylated alcohols and fatty acid ester sulphonates, especially C_8 to C_{22} fatty acid C_1 to C_6 esters.

Any phosphate employed in the compositions of this invention will function as a detergent builder. Other
25 detergent builder may also be included, but preferably it should not raise the level of the phosphorus beyond the level

of 5% by weight. Buffer materials to control the pH of the liquor formed when the detergent laundry bar is rubbed onto wet fabric may also be included. However, for the low cost bars of this invention the levels of detergent builder and
5 buffer may be low. Apart from phosphate, the preferred builder and buffer material is sodium carbonate. /

Typical fillers which may be employed are calcite, various types of clays (e.g. kaolin and bentonite) and sodium sulphate, the amounts and choice of which are adaptable to
10 volume and economical considerations.

Other ingredients, such as starch, sodium carboxymethylcellulose, colouring materials, fluorescers, opacifiers, germicides, perfumes, including deoperfumes, etc, may also be incorporated as desired.

15 The laundry bar of the invention can be and is preferably prepared by a process, e.g. in a high shear "Z" blade mixer, comprising the steps of neutralising the anionic-active acid, e.g. alkyl benzene sulphonate acid, with alkali, e.g. sodium carbonate or with alkaline silicate,
20 adding all or the major part of the formulation water during or after neutralisation, followed by mixing therewith the appropriate solids to form a dough and adding any sensitive and minor ingredients at the final mixing stage.

Heat is normally generated during the
25 neutralisation, by hydration and by the high shear rates, but, if not sufficient, heating may be applied, e.g. to

adjust the temperature to about 60-65°C.

Example 1

Laundry bars were prepared with the compositions /
set out in Table 1 below. The ingredients were mixed into
5 doughs using a 1Kg "Z" blade mixer and then 1 inch (25cm)
square billets were extruded using a 2 inch (50cm) diameter
single screw plodder.

The bars were stored for 7 days and then ^{1cs} ~~samples~~ of
each bar were immersed for 4 days in toluene. Inspection of
10 the bars after this treatment revealed whether the bars had a
silicate-based continuous phase (in which case the bars
retained structural integrity) or a continuous phase based on
the detergent active in which case the bars disintegrated.
Some bars displayed an intermediate state, swelling, cracking
15 and reaching a fragile, crumbling condition.

The hardness of bars stored for 7 days was measured
with a penetrometer - using a 200gm load applied to a
standard needle.

The rate of wear of bars which had been stored for
20 7 days was assessed as the weight loss when the bars were
rubbed 500 times over wet fabric, using a mechanical rig.

Bars which had been stored for 7 days were
subjected to immersion in water for two hours, after which
soft mush was scraped from the bar. The weight loss and the
25 weight of scraped-off mush were noted.

TABLE 1

% Ingredient (Nominal, by weight)		A	B	C	D	E	F	G	H
5	Sodium linear alkyl benzene sulphonate	20	12	28	20	20	20	24	20
	Sodium alkaline silicate	15	15	15	5	10	22	15	6.7
10	Trisodium orthophosphate	5	5	5	5	5	5	5	5
	Calcite	40.0	39.7	35.0	48.0	48.7	32.0	36.0	46.0
	Water	18.5	18.5	18.5	12.0	14.0	18.5	16.25	11.5
	Fluorescent agent	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
15	Perfume	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
	Blue dye	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
	Non-detergent organic matter	----- balance -----							

Results of the toluene immersion test, bar hardness
 and rate of wear are set out in Table 2 below. Results of
 the toluene immersion test are also shown in graph form in
 Figure 1.

TABLE 2

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Bar	A	B	C	D	E	F	G	H
Continuous phase	S	S	A	A	S	S	I	I
5 (S = silicate A = detergent active I = intermediate)								
Penetration (mm)	1.9	1.1	10.5	3.5	1.2	1.4	4.5	
10 Rate of wear				51.7	23.1	22.8		30.1

As can be seen from Table 2, the bars with a silicate-based continuous phase display a good level of hardness and rate of wear, both of which are fairly constant despite variation in the silicate level.

15 Example 2

Bar samples were made generally as in Example 1, but with and without silicate and with and without orthophosphate. Mixing order was the same in each case. Penetrometer tests with similar test conditions were carried out on fresh samples and samples which had been stored for one day. Partial compositions and results are set out in Table 3 below:

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TABLE 3

% Ingredients				
	Sodium linear	20	20	20
	alkyl benzene sulphonate			
5	Sodium alkaline silicate	10	-	10
	Trisodium orthophosphate	5 "	5	-
	Hardness when fresh	2.4	9.4	12.7
	(mm penetration)			
	Hardness after 1 day	0.7	5.6	4.5
10	(mm penetration)			

Thus orthophosphate displays greater hardening effect in the presence of silicate than when silicate is absent.

Commercial bars with a detergent-based continuous
 15 phase and fairly high levels of phosphate present would generally give a penetration depth of less than 1mm after storage for one day.

What we claim is:

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1. A laundry bar including a non-soap detergent active compound and an alkali metal silicate, wherein the
5 detergent active is from 10 to 35% by weight of the composition, the silicate is from 5 to 20% by weight of the composition, and their relative proportions give the bar an inorganic, silicate-containing continuous phase, whereby the bar retains its structural integrity on
10 immersion in toluene for 4 days at 20°C.

2. A laundry bar according to claim 1 additionally comprising an inorganic filler.

3. A laundry bar according to claim 1 additionally comprising an inorganic electrolyte which is a
15 hydratable salt, or the hydrate form of such a salt.

4. A laundry bar according to claim 3 wherein the salt
20 is a trialkali metal orthophosphate or a penta alkali metal tripolyphosphate with a high phase I content.

5. A laundry bar according to claim 4 containing the phosphate in an amount such that the phosphorus content
25 of the bar is no more than 1.5% by weight.

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6. A laundry bar according to claim 3 wherein the salt is an alkali metal carbonate, bicarbonate or sesquicarbonate.
- 5 7. A laundry bar according to ^{claim 1} ~~any one of the preceding~~ ^{See P-13} ~~claims~~ wherein the amount of the non-soap detergent active compound is from 10 to 25% by weight.
- 10 8. A laundry bar according to claim 1 including a phosphate salt in an amount of no more than 5% by weight and the silicate salt in an amount of at least 6% by weight.
- 15 9. A laundry bar according to claim 8 including the silicate salt in an amount of at least 8% by weight.

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ABSTRACT

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suitable for hand washing of fabrics. Conventionally such
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