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(71) Applicant: **UNILEVER PLC**
Unilever House Blackfriars
London EC4P 4BQ(GB)
(84) **GB**

Applicant: **UNILEVER NV**
Burgemeester s'Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam(NL)
(84) **CH DE ES FR IT LI NL SE**

(72) Inventor: **Thompson, Laurence Griffith**
34 South Drive Upton
Wirral Merseyside L49 6LA(GB)
Inventor: **Hull, Michael**
Windcliffe Alvanley Road
Helsby Warrington WA6 9PS(GB)
Inventor: **Giles, Dennis**
33 Brookhurst Road Bromborough
Wirral Merseyside L63 0EH(GB)
Inventor: **Hammond, Estelle Jacqueline**
1 Porto Hey Road Irby
Wirral Merseyside L61 2XA(GB)
Inventor: **Bernardi, Paolo**
Via Sassi 6
I-20090 Vimodrone(IT)

(74) Representative: **Ford, Michael Frederick et al**
MEWBURN ELLIS 2 Cursitor Street
London EC4A 1BQ(GB)

(54) Detergent compositions.

(57) A detergent composition for washing soiled materials, notably fabrics is based on alkyl orthoxylene sulphonate as detergent active. Good detergency is achieved by restricting electrolyte concentration so that there is an ionic strength of not more than 0.05 mole/litre in the wash liquor. A detergent composition for this purpose contains not more than 60% by weight of water-soluble alkali metal salts and preferably contains zeolite as water-insoluble builder. Alternatively or additionally a second surfactant, which is soluble in electrolyte solution is included, in which case ionic strength may be greater than 0.02 mole/litre and there is synergy between the surfactants.

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DETERGENT COMPOSITIONS

This invention relates to detergent compositions, particularly but not exclusively to detergent compositions for cleaning fabrics.

Detergent compositions generally comprise a number of ingredients, including a detergent active material to assist the removal of soil from the materials to be cleaned and the suspension of the removed soil in the wash liquor. Notable applications of detergent compositions are to clean fabrics usually by washing portable fabric items in a bowl or in a washing machine, to clean hard surfaces such as glass, glazed surfaces, plastics, metals and enamels and to clean soft furnishings such as carpets. A number of classes of materials have been used as detergent active materials, including anionic and nonionic materials. Typical anionic materials are the alkyl benzene sulphonates.

Detergent active materials are useful not only in the washing of fabrics but in many other fields, such as in the oil industry. Among the numerous materials which have been proposed for use in the oil industry as detergent active materials are the alkyl orthoxylene sulphonates. Thus, US 3861466 (Exxon/Gale) discloses the recovery of crude oil by injecting alkyl orthoxylene sulphonate into a subterranean formation. However, attempts to use such materials as detergent active materials in fabric washing products showed that they were not especially beneficial and they have not therefore been used for fabric washing nor any other domestic application.

However, we have now discovered that alkyl orthoxylene sulphonates can be used as effective detergent active materials in detergent products under certain conditions and that under these conditions the performance of such materials may even surpass more conventional detergent active materials.

Thus according to a first aspect of this invention there is provided a method of washing which comprises contacting soiled materials with a wash liquor which includes:

at least 0.05 g/l of one or more surfactants comprising an alkyl orthoxylene sulphonate; and wherein

i) the ionic strength in the wash liquor is not more than 0.05 mole/l, or

ii) the surfactants include an anionic or nonionic second surfactant which is sufficiently soluble as to be able to give a clear solution of at least 0.5 gram of surfactant per litre in 0.1 molar aqueous sodium chloride solution, the ratio by weight of the alkyl orthoxylene sulphonate to the second surfactant lying in the range from 1:20 to 9:1.

It is particularly envisaged that the soiled material will be fabric but other possibilities include dishes, other hard surfaces and soft furnishings.

The amount of surfactant in the wash liquor is preferably from 0.1 g/l or 0.25 g/l up to 1.5 g/l. It may be higher, e.g. up to 2.5 g/l but is unlikely to exceed 5.0 g/l. Preferably there is at least 0.05 g/l of alkyl orthoxylene sulphonate even when this is not the sole surfactant.

Both (i) and (ii) above are conditions under which alkyl orthoxylene sulphonate may beneficially be used for washing soiled materials. These conditions of use are not mutually exclusive, although when a second surfactant of the kind specified above is employed, it can be advantageous for the wash liquor to have an ionic strength of at least 0.02 moles/litre. However, a second surfactant, possibly or possibly not of the specified solubility, may be used below this ionic strength.

Ionic strength is related to concentration but takes account of the numbers of ions in a molecule and multiple charged ions.

Ionic strength is calculated from the molarity (m) of each ionic species present in solution and the charge (z) carried by each ionic species. Ionic strength (I) is one half the summation of $m.z^2$ for all ionic species present i.e.

$I = \frac{1}{2} \Sigma m.z^2$

For a salt whose ions are both univalent, ionic strength is the same as the molar concentration. This is not so where more than two ions or multiple charges are involved. For instance a 1 molar solution of sodium carbonate contains 2 moles/litre of sodium ions and 1 mole/litre of carbonate ions carrying a double charge. Ionic strength is given by:

$I = \frac{1}{2} [2(1^2) + 1 \times (2^2)] = 3$ moles/litre

A description of ionic strength is given in "Physical Chemistry" by Walter J. Moore, 4th Ed. 1963.

It may be the case that both ionic strength and electrolyte concentration will lie on the same side of a critical value, for example both below 0.05 moles/litre or both above 0.02 moles/litre.

An ionic strength of 0.05 moles/litre or less would be a relatively low level of electrolyte, for a liquor to be used in fabric washing where soluble salts are generally included in the detergent composition which is used.

The level of electrolyte in the wash liquor is not, in practice, a parameter over which the domestic user of a detergent product exercises direct control. It is determined, inter alia by the level of water-soluble salts present in the product and the recommended dosage for that product.

Thus forms of the invention with an ionic strength in the wash liquor of not more than 0.05 mole/litre are of particular value where (i) the product has a low content of inorganic material, e.g. some formulations for hand dishwashing, (ii) where recommended dosage levels are low, as for example in North America, or (iii) where the product contains high levels of water-insoluble material, as for example where a fabric washing product contains a water-insoluble detergency builder material. The only water-insoluble detergency builder material which has been used extensively is zeolite, a crystalline aluminosilicate ion-exchange material, but the invention would also be of value to products containing any other water-insoluble builder material.

Detergent compositions are normally used at a dosage exceeding 1g/l (which would be an exceptionally low level of usage). Dosage is typically in the range 4 g/l to 10m g/l. A composition designed to yield an ionic strength of not greater than 0.05 moles/litre will generally not contain more than 60% of water-soluble alkali metal salts.

Certain useful forms of such compositions also contain from 10% to 60% preferably from 20% to 50% by weight of a water-insoluble detergency builder material.

Thus, in a second aspect, this invention provides a detergent composition comprising: from 1% to 50% by weight of a detergent active material which comprises an alkyl orthoxylene sulphonate; which composition:

i) does not contain more than 60% by weight of water-soluble alkali metal salt, or

ii) includes in the detergent active material an anionic or nonionic second surfactant which is sufficiently soluble as to be able to give a clear solution of at least 0.5 gram of surfactant per litre in 0.1 molar aqueous sodium chloride solution, the ratio by weight of the alkyl orthoxylene sulphonate to the second surfactant lying in the range from 1:20 to 9:1.

Compositions intended to provide an ionic strength below 0.05 moles/litre, as mentioned above, may include a second surfactant of the solubility specified above, especially of course for ionic strengths in the range 0.02 to 0.05 moles/litre.

Further surfactant, whether or not of the specified solubility, may possibly be included, preferably at a level no more than that of the alkyl orthoxylene sulphate. Indeed the amount of such further surfactant may be confined to not over 50% or even not over 10% of the amount of orthoxylene sulphonate.

At particularly low ionic strengths, synergistic action is observed between alkyl orthoxylene sulphonate and nonionic surfactant with a low HLB. Such mixtures may have more of the nonionic surfactant than of the alkyl orthoxylene sulphonate, e.g. lying within a range of ratios from 1:7 to 7:1.

As mentioned above, forms of the invention which contain a second surfactant may utilise an ionic strength of at least 0.02 mole/litre. At such ionic strength, alkyl orthoxylene sulphonate is rather insoluble, and on its own gives turbid liquors at useful surfactant concentrations.

Suitable second surfactants should have adequate solubility to dissolve in the wash liquor at the time of use. This signifies sufficient solubility so that, if used alone, they give a clear solution in electrolyte of the envisaged ionic strength of 0.02 mole/litre or more. For convenience this solubility is here defined as the ability to dissolve to a clear solution in 0.1 molar sodium chloride.

Mixtures of alkyl orthoxylene sulphonate with a second surfactant having the specified solubility generally display synergy over a range of proportions. This synergy may be apparent as a degree of detergency greater than that achieved by either surfactant used alone. Alternatively it may be a degree of detergency which is not quite as high as that of one of the surfactants if used at the same total surfactant level, but which is better than would be predicted by interpolation between detergency values observed for the two surfactants. This lesser synergy, which does not surpass the detergency of one of the two surfactants may well be useful for other reasons. For instance, incorporation of some anionic surfactant makes it easier to produce particulate compositions with adequate structural strength. Slight loss in detergency as compared with using only a nonionic surfactant would therefore be tolerable. Also, the detergency of compositions based on nonionic surfactants alone tends to be affected by residues left from previous washes. Incorporation of anionic surfactant reduces this tendency, making compositions more robust. Consequently, if synergy between alkyl orthoxylene sulphonate and a nonionic surfactant improves their detergency to approach that of the nonionic alone, there is an overall benefit.

The proportions of alkyl orthoxylene sulphonate and a second surfactant which give best results will depend on the identity of the second surfactant used, and on the electrolyte concentration in the wash liquor. Moreover the electrolyte concentration at which the detergency can be optimised will also depend on the identity of the second surfactant. Optimum proportions and optimum electrolyte concentration can be investigated by trials of the kind set out below as examples.

In order to provide an ionic strength of at least 0.02 mole/litre in the wash liquor it is suitable to include at least 15% by weight better at least 25% and perhaps 40% or more of water-soluble salts in the detergent composition. The amount of water-soluble salts may extend up to 80%, possibly even 90 or 95% of the detergent composition. If the level of electrolyte is towards the lower (i.e. 15%) end of the range the user may need to use the composition at levels approaching or even exceeding 10 gram/litre. It is not normally convenient for the electrolyte concentration in a wash liquor to exceed 0.5 molar.

It follows from the percentages just stated that compositions containing a soluble second surfactant and intended to provide an ionic strength exceeding 0.02 mole/litre may be built with water-soluble phosphate, for example sodium tripolyphosphate, or may be built with an insoluble builder provided this is accompanied by other water-soluble salts.

The alkyl orthoxylene sulphonates of the present invention are in general based on alkyl xylenes having an average molecular weight of at least 295, usually 300 to 390 and preferably 305 to 360. The alkyl side chain generally lies within a range from about C_{12} to C_{24}, preferably having an average chain length of 15 to 20 carbon atoms. The alkyl side chain may be substantially all branched alkyl, when it is preferred that not more than 60% (preferably not more than 40%) of the branched alkyl groups have the aromatic ring at the 2 position of the alkyl chain, the remainder of the alkyl groups being at the 3 position or even further along the alkyl chain. The alkyl xylene is normally prepared by the alkylation of xylene with an appropriate olefin. The alkylate so produced is then sulphonated and neutralised in known manner. The solubilising cation of the neutralised product may be alkali metal, ammonium or substituted ammonium. The alkyl orthoxylene sulphonates of this invention are distinct from orthoxylene sulphonate (without an alkyl group) which is known to be useful as a hydrotrope.

Anionic surfactants which may be used as the second surfactant of specified solubility are alkali metal, ammonium and alkylolammonium (e.g. monoethanol ammonium, diethanol ammonium, triethanol ammonium) salts of organic acids containing carboxylic, sulphonic or sulphuric acid groups. Examples of such anionic materials suitable as co-surfactants with alkyl orthoxylene sulphonates are as follows. C_8-C_{18} alkyl benzene sulphonates, C_8-C_{18} alkyl or alkenyl sulphates, preferably C_{12}-C_{16} alkyl sulphates, most preferably C_{12}-C_{15} alkyl α alkyl sulphates where the branched chain in the α position may be methyl, ethyl, propyl or butyl. Longer branches may occur in the α position providing such material is of less than 10% of the total alkyl sulphate. C_8-C_{18} alkoxy sulphates wherein the alkoxy group is preferably ethylene oxy and contains from 2-5 most preferably 2-3 ethylene oxy groups. Esters of α-sulphonated fatty acids from C_{12}-C_{20} preferably C_{16}-C_{18} methyl esters, most preferred are C_{12}-C_{18} esters of ethyl, propyl butyl. C_{12}-C_{18} alkyl or alkenyl straight or branched chain alkyl glycerol ether sulphonates and sulphates, C_{12}-C_{18} alkyl monoglyceride sulphonates and sulphates. C_{10}-C_{20} alkyl or alkenyl carboxylates, or α-hydroxy carboxylates, C_8-C_{18} α-olefin sulphonates, C_6-C_{18} internal olefin sulphonates, C_6-C_{18} vinylidene olefin sulphonates. Dialkyl sulpho-succinates of total chain length of C_{12}-C_{16} where only one chain may be longer than six carbon atoms. Sulphates from Guerbet alcohols wherein the alkyl chains are C_{12}-C_{16} where only one chain may be greater than six carbon atoms. C_{12}-C_{16} di alkoxy propyl sulphates where only one chain may be greater than six carbon atoms. The salts of the reaction products of fatty acids from C_{12}-C_{18} both saturated and unsaturated with isethionic acid. Secondary alkyl sulphonates of alkyl chain length C_8-C_{20} where the alkyl chain may be branched and contain some unsaturation. Secondary alkyl sulphates where the alkyl chain length is C_8-C_{20} and the sulphate group is such that of the two alkyl chains only one is longer than six carbon atoms.

Suitable soluble nonionic surfactants have an HLB greater than 10.5. Examples of such nonionics are as follows. Polyethylene oxide condensates of alkyl alcohols of C_8-C_{20} alkyl chains with an ethylene oxide content from 2 to 20 moles per mole of alkanol. The alkyl chains may be straight or branched and may include unsaturation. Polyethylene oxide condensates of alkyl phenols having an alkyl chain from 6-12 carbon atoms in a straight or branched chain configuration with ethylene oxide content from 2 to 20 moles of ethylene per mole of alkyl phenol. Polyethylene oxide condensates of secondary alkyl alcohols of chain length C_6-C_{20} with 2-20 moles of ethylene oxide per mole of secondary alcohol. Polyethylene oxide condensates of C_8-C_{20} fatty acid alkylolamides in which each alkylol group contains from 1-3 carbon atoms and can contain from 2-20 ethylene oxide groups per mole of alkylolamide. In all the polyethylene oxide condensates it is preferable if the ethylene oxide distribution is narrow with at least less than 5% free alkanol. An alkyl polyglycoside of the following generic type:

$R' O(C_nH_{2n}O)_t (Z)_x$

where R' is alkyl, alkyl phenyl or mixtures and the chain may be saturated, unsaturated, branched or unbranched from C_6-C_{20} and may have up to three hydroxy groups in the chain. the polyalkoxide moiety is preferably ethoxide but can be propoxide, i.e. n is 2 or 3. The content of polyalkoxide is from 0 to 10 units (t = 0-10) preferably 0. The alkyl polyglycoside contains a reducing sugar (Z). The reducing sugar can be either hexose or pentose sugars. The degree of polymerization (x) can extend up to 10 preferably from 1.5

to 3. Individual alkyl polyglycosides will have x as an integer. However, mixtures can be used in which case x is an average degree of polymerization. The alkyl chain can be attached at the 1-,2,-3,-4,- or 6-carbon atoms. The preferred attachment is via the 1-carbon atom.

Electrolyte material which is present will generally be a water-soluble salt. This may be a salt such as will provide a benefit in terms of the fabric washing process such as a water-soluble detergency builder salt or a water-soluble alkaline salt. Alternatively the electrolyte may be constituted by a water-soluble filler salt such as sodium sulphate or sodium chloride. Salts from all three classes may be present. It is unlikely that water-soluble salts will be entirely absent. In practice they will generally provide an ionic strength exceeding 10^{-3} moles/litre and will constitute more than 1% of a detergent composition.

Examples of phosphorus-containing inorganic water-soluble detergency builder salts, include the alkali metal pyrophosphates, orthophosphates, polyphosphates and phosphonates. Specific examples of inorganic phosphate builders include sodium and potassium tripolyphosphates, orthophosphates and hexametaphosphates.

Examples of non-phosphorus-containing water-soluble inorganic detergency builder salts, include alkali metal carbonates and bicarbonates. Specific examples include sodium carbonate, potassium carbonate and sodium and potassium bicarbonates (which may be accompanied by calcite seed crystals).

Examples of water-soluble organic detergency builder salts include the alkali metal, ammonium and substituted ammonium polyacetates, carboxylates, polycarboxylates, polyacetyl carboxylates and polyhydroxysulphonates.

Specific examples include sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylenediaminetetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, melitic acid, benzene polycarboxylic acids and citric acid.

It is preferred that the compositions be alkaline, that is at a concentration of 1 g/l or more in distilled water at 25° C, the pH should be at least 8, preferably at least 10. If any water-soluble builder salt which is present is insufficient to achieve this, a water-soluble alkaline material such as the alkali metal (especially sodium) silicates may be included.

The water-insoluble detergency builder materials may be selected from aluminosilicate builder materials, either their amorphous or crystalline forms, such as zeolite A and seed crystal materials such as high surface area calcite which will normally be used in conjunction with a water-soluble carbonate salt such as sodium carbonate.

As mentioned above, apart from surfactant and builder materials, a number of optional ingredients may also be present.

Examples of other ingredients which may be present in the composition are polymers containing carboxylic or sulphonic acid groups in acid form or wholly or partially neutralised to sodium or potassium salts, the sodium salts being preferred. Preferred polymers are homopolymers and copolymers of acrylic acid and/or maleic acid or maleic anhydride. Of especial interest are polyacrylates, acrylic/maleic acid copolymers, and acrylic phosphinates.

Suitable polymers, which may be used alone or in combination, include the following:

polyacrylic acids, for example Versicol (Trade Mark) E5, E7 and E9 ex Allied Colloids, Narlex (Trade Mark) LD 30 and LD 34 ex National Adhesives and Resins Ltd, Acrysol (Trade Mark) LMW-10, LMW-20, LMW-45 and Al-N ex Röhm & Haas, and Sokalan (Trade Mark) PA-20, PA-40, PA-70 and PA-110 ex BASF;

ethylene/maleic acid copolymers, for example the EMA (Trade Mark) series ex Monsanto;

methyl vinyl ether/maleic acid copolymers, for example, Gantrez (Trade Mark) AN 119 and AN 149 ex GAF Corporation;

acrylic acid/maleic acid copolymers, for example, Sokalan (Trade Mark) CP4, CP5 and CP7 ex BASF, and the Alcosperse (Trade Mark) series ex Alco;

acrylic phosphinates, for example, DKW (Trade Mark) 125 ex National Adhesives and Resins Ltd, and the Belsperse (Trade Mark) series ex Ciba-Geigy.

The molecular weights of homopolymers and copolymers are generally 1000 to 150,000, preferably 1500 to 100,000. The amount of any polymer may lie in the range from 0.5 to 5% by weight of the composition. Other suitable polymeric materials are cellulose ethers such as carboxy methyl cellulose, methyl cellulose, hydroxy alkyl celluloses, and mixed ethers, such as methyl hydroxy ethyl cellulose, methyl hydroxy propyl cellulose, and methyl carboxy methyl cellulose. Mixtures of different cellulose ethers, particularly mixtures of carboxy methyl cellulose and methyl cellulose, are suitable. Polyethylene glycol of molecular weight from 400 to 50,000, preferably from 1000 to 10,000, and copolymers of polyethylene oxide with polypropylene oxide are suitable as also are copolymers of polyacrylate with polyethylene glycol. Polyvinyl pyrrolidone of molecular weight of 10,000 to 60,000 preferably of 30,000 to 50,000 and copolymers of polyvinyl pyrrolidone with other poly pyrrolidones are suitable. Polyacrylic phosphonates and related copolymers of molecular weight 1000 to 100,000, in particular 3000 to 30,000, are also suitable.

Further examples of other ingredients which may be present in the composition include fabric softening agents such as fatty amines, fabric softening clay materials, lather boosters such as alkanolamides, particularly the monoethanolamides derived from palm kernel fatty acids and coconut fatty acids, lather depressants, oxygen-releasing bleaching agents such as sodium perborate and sodium percarbonate, peracid bleach precursors, chlorine-releasing bleaching agents such as trichloroisocyanuric acid, inorganic salts such as sodium sulphate, and, usually present in very minor amounts, fluorescent agents, perfumes including deodorant perfumes, enzymes such as cellulases, proteases and amylases, germicides and colourants. Hard surface cleaners will contain abrasives such as calcite or silica and may contain organic solvent.

If such other ingredients are in the form of water-soluble salts, it may well be necessary to restrict the use of them, if it is intended to maintain the ionic strength in the wash liquor below a critical level, such as 0.05 moles/litre. Alternatively, measures may be taken to suppress the dissolution of such ingredients at least until part way into the wash process.

The detergent compositions according to the invention may be in any suitable form including powders, bars, liquids and pastes. For example suitable liquid compositions may be non-aqueous or aqueous, the latter being either isotropic or lamellar structured. The compositions may be prepared by a number of different methods according to their physical form. In the case of granular products they may be prepared by dry-mixing or coagglomeration. A preferred physical form is a granule incorporating a detergency builder salt and this is most conveniently manufactured by spray-drying at least part of the composition. In this process a slurry is prepared containing the heat-insensitive components of the composition such as the surfactant system, builder material and filler salt. The slurry is spray-dried to form base powder granules with which any solid heat-sensitive ingredients may be mixed, such ingredients including bleaches and enzymes.

The invention will now be described in more detail in the following non-limiting Examples in which Examples 1 to 6 demonstrate use at ionic strengths below 0.05 mole/litre and Examples 7 onwards demonstrate the effect of a soluble second surfactant. In the Examples washing experiments were carried out using a standard procedure.

Wash liquors were prepared containing one or more surfactants, as stated. The total surfactant concentration was in the region of 1.0 gram/litre. Such a concentration would be achieved using 6 gram/litre of a product containing approximately 16.5% by weight of surfactant.

400ml of wash liquor was used to wash 10g of knitted polyester test cloth soiled with 2.4% of radiolabelled hexadecane or triolein. (Thus the liquor:cloth ratio was 40:1). Soil removal was monitored by measuring the content of the radiolabelled substance in the wash liquor. Washing was carried out in a Tergotometer at 70 oscillations per minute.

EXAMPLE 1

This Example demonstrates the effect of electrolyte ionic strength on the detergency of hexadecane soil from fabrics by the use of secondary C_{16} alkyl ortho xylene sulphonate. This had a distribution of alkyl chain lengths which included 80% C_{16}, 9% C_{13} and 8% C_{14}. The distribution of positions at which the aromatic ring was substituted on the alkyl group was 29% 2 position, 26% 3 position, 14% 4 position, 31% 5 position or beyond.

The wash liquors were prepared so as to contain 1.0 g/l of the alkyl orthoxylene sulphonate and varying concentrations of sodium chloride as electrolyte. The wash temperature was 40°C. The soil on the test cloths was C^{14} labelled hexadecane. The results are set out in the following Table.

NaCl Level (mole/l)	Soil Removal (%)
0.01	33
0.02	45
0.0355	17
0.05	5
>0.05	0

These results demonstrate that with increasing electrolyte in the wash liquor, the soil removal at first increases, but when an electrolyte level of 0.0355 mole/l has been reached, the performance has begun to fall off rapidly to unacceptable levels.

EXAMPLE 2

The procedure of Example 1 was repeated, but on this occasion comparing the performance of the same C_{16} alkyl orthoxylene sulphonate with that of a conventional C_{16} alkyl benzene sulphonate. The test cloths were made from a different batch of polyester to that used for Example 1. The soil was removed to a greater extent than with the cloths of Example 1, but this does not affect the relative values observed. Results were as follows:

	Soil Removal (%)	
NaCl Level (mole/l)	alkyl o-xylene sulphonate	alkyl benzene sulphonate
5×10^{-3}	35	22
0.01	62	47
0.02	67	56
0.03	41	59
0.06	11	65
0.12	3	71
0.3	0	56

These results clearly demonstrate that, at low ionic strengths below about 0.03 mole/l the alkyl orthoxylene sulphonate gives superior performance. At higher electrolyte levels, the more usually employed alkyl benzene sulphonate is superior.

EXAMPLE 3

A wash liquor was prepared with an ionic strength of 0.023 mole/l corresponding to use of 6 g/l of a product containing the following constituents:

	% by weight
Alkyl orthoxylene sulphonate (as in Example 1)	13
Zeolite	24
Alkaline silicate	3
Polyacrylate	4
Sodium carbonate	6
Sodium sulphate	10
Water, and other constituents which are not electrolytes	balance

This formulation would be typical for a product which was a detergent powder with zeolite as builder. The pH of the wash liquor was approximately 10.

The soil on the polyester test cloths was C^{14} labelled triolein. The was temperature was again 40° C. A comparative wash liquor, with C_{12} alkyl benzene sulphonate, in place of alkyl orthoxylene sulphonate, was used in the same way. The amount of triolein removed was 39% when the detergent active was the alkyl orthoxylene sulphonate, contrasting with 29% when the detergent active was C_{12} alkyl benzene sulphonate.

The ionic strength in the wash liquor containing alkyl orthoxylene sulphonate was varied by varying the concentration of sodium carbonate, which in turn varied the pH slightly (between 9.6 and 10.4). Maximum detergency (i.e. greatest soil removal) was observed when the ionic strength was 0.03 mole/l.

EXAMPLE 4

A wash liquor was prepared with an ionic strength of 0.01 mole/l corresponding to use of 7.7 g/l of a liquid product containing the following constituents:

	% by weight
Alkyl orthoxylene sulphonate (as in Example 1)	13.0
Glycerol	4.0
Borax	1.3
Sodium citrate	7.0
Sodium chloride	2.32
Ethanol	30.0
Water and minor constituents which are not electrolytes	balance

The pH of the wash liquor was approximately 9. The polyester test cloths were again soiled with C^{14} labelled triolein and washed at 40° C as in previous Examples. A comparative wash liquor with C_{12} alkyl benzene sulphonate in place of alkyl orthoxylene sulphonate was used in the same way. Triolein removal was:

alkyl orthoxylene sulphonate 48.0%
alkyl benzene sulphonate 17.1%

EXAMPLE 5

Example 4 was repeated without sodium chloride but with sufficient sodium hydroxide to increase the pH to 10. This ionised the sodium citrate fully and again yielded an electrolyte concentration of 0.01 mole/l. Triolein removal was:

alkyl orthoxylene sulphonate 45.5%
alkyl benzene sulphonate 15.9%

EXAMPLE 6

Wash liquors were prepared containing 1.0 g/l of surfactant which was the C_{16} alkyl orthoxylene sulphonate used in previous examples, or a nonionic surfactant, or a mixture of the two. The nonionic surfactant was C_{12} alcohol ethoxylated with an average of 3 ethylene oxide residues and having an HLB value of 8.1.

The wash liquors contained sodium chloride and sodium carbonate to give varying ionic strengths, and a pH of 10.

The polyester test cloths were soiled with C^{14} radiolabelled triolein. The wash temperature was 25° C. Triolein removal at the various ionic strengths was as set out in the following Table:

		% triolein removal at stated ionic strength			
% alkyl orthoxylene sulphonate	% nonionic	0.005 mole/l	0.01 mole/l	0.02 mole/l	0.03 mole/l
0	100	7	7	7	7
20	80	17	16	15	14
40	60	12	15	15	15
60	40	9	13	15	16
80	20	9	12	16	19
100	0	8	12	24	25

It can be seen that at ionic strengths of 0.005 and 0.01 mole/l there is synergy between this low HLB nonionic and the alkyl orthoxylene sulphonate. The triolein removal by mixtures was equal to or exceeded that achieved by either surfactant alone.

EXAMPLE 7

C_{16} alkyl orthoxylene sulphonate as in previous Examples was used in conjunction with a nonionic surfactant. This was fatty alcohol of average chain length C_{12}, ethoxylated with an average of 6 ethylene oxide residues. The HLB value of this nonionic surfactant was 11.8.

Wash liquors were prepared so as to contain the surfactants in varying proportions, but always with a total surfactant concentration of 1 g/l. The wash liquors contained sodium chloride to give an electrolyte level of 0.05 mole/litre. The polyester test cloths were soiled with C^{14} labelled hexadecane. The wash temperature was 25° C.

The results of the washing experiments are set out in the following Table:

Surfactant Ratio	
C_{16} alkyl o-xylene sulphonate : C_{12} 6EO	% Hexadecane Removal from polyester
0 : 100	62
10 : 90	63
25 : 75	70
37.5 : 62.5	63
50 : 50	52
55 : 45	52.5
60 : 40	65
65 : 35	60
67.5 : 32.5	48
70 : 30	13
75 : 25	5
100 : 0	0

Synergy between the two surfactants is apparent. Maximum soil removal occurred at a 1:3 ratio, but over a range of ratios soil removal was greater than the value which would be predicted by interpolation between the end points.

EXAMPLE 8

The same C_{16} alkyl orthoxylene sulphonate was used in conjunction with a different nonionic surfactant. This was fatty alcohol of average chain length C_{12} ethoxylated with an average of 8 ethylene oxide units per molecule.

This nonionic surfactant had an HLB value of 13:1. Wash liquors contained a total surfactant concentration of 1 g/l as in Example 7.

Several ionic strengths were used. Ratios of surfactants, ionic strengths and triolein removal are set out in the Table below.

		% Triolein removal at stated ionic strengths			
% Alkyl o-xylene Sulphonate	% C_{12}8EO Nonionic	0.1 mole/l	0.05 mole/l	0.03 mole/l	0.01 mole/l
0	100	29	29	29	29
20	80	24	22	14	5
40	60	28	21	10	4
60	40	39	37	24	4
80	20	22	37	34	5
100	0	7	17	22	7

It can be seen from this Table that at an ionic strength of 0.01 mole/l, the triolein removal is no better with mixed surfactants than with orthoxylene sulphonate alone. In contrast, at higher electrolyte levels there is a manifest synergy.

EXAMPLE 9

Example 8 was repeated, using the same combination of surfactants as in Example 7. Ratios of surfactants, ionic strengths and the triolein removal are set out in the Table below.

		% Triolein removal at stated ionic strengths			
% Alkyl o-xylene Sulphonate	% C_{12}6EO Nonionic	0.03 mole/l	0.05 mole/l	0.07 mole/l	0.1 mole/l
0	100	64.2	66.6		68.6
20	80	35.0	46.0	42.4	68.0
40	60	26.2	40.5	41.1	66.0
60	40	27.6	32.9	28.2	36.0
80	20	14.4	13.4	19.7	7.8
100	0	11.2	10.9	19.0	6.2

As a comparison, an alkyl benzene sulphonate, with average C_{12} alkyl chain length was used with the same nonionic surfactant at 0.1 mole/l ionic strength. The results were:

		% Triolein removal	
%anionic	%nonionic	alkyl o-xylene sulphonate with C_{12}6EO	alkylbenzene sulphonate with C_{12}6EO
0	100	69	69
20	80	68	54
40	60	66	45
60	40	36	45
80	20	8	44
100	0	6	41

It is apparent that there is synergy between the alkyl orthoxylene sulphonate and the nonionic surfactant. By contrast with the results for 0.1 mole/l electrolyte in the previous Example, triolein removal by mixed surfactants is better than by orthoxylene sulphonate alone. Secondly, although the results for mixed surfactants are inferior to those for nonionic surfactant alone, at 20% anionic and 40% anionic, the triolein removal with the alkyl orthoxylene sulphonate was superior to that with alkyl benzene sulphonate.

EXAMPLE 10

The same C_{16} alkyl orthoxylene sulphonate was used in admixture with each of several other surfactants, in each case at an ionic strength of 0.1 mole/l. The other surfactants were:
Sodium dodecyl sulphate (SDS)
C_{16} internal olefin sulphonate (IOS)
C_{16} alpha olefin sulphonate (AOS) Nonyl phenol, ethoxylated with average 10 ethylene oxide residues per molecule (NP-10EO).

Triolein removal values are set out in the following Table.

	% Triolein removal			
% alkyl o-xylene Sulphonate	SDS	IOS	AOS	NP-10EO
0	37	10	33	62
20	42	16	33	62
40	42	13	39	59
60	40	9	37	57
80	25	8	35	17
100	7	7	7	7

Synergy between the two surfactants is apparent in each instance. With the nonionic surfactant, the result for nonionic alone matches the triolein removal for 20% orthoxylene sulphonate 80% nonionic. The situation is analogous to that in the previous Example.

Claims

1. A method of washing which comprises contacting soiled materials with a wash liquor which includes: at least 0.05 g/l of one or more surfactants comprising an alkyl orthoxylene sulphonate; and wherein

i) the ionic strength in the wash liquor is not more than 0.05 mole/l, or ii) the surfactants include an anionic or nonionic second surfactant which is sufficiently soluble as to be able to give a clear solution of at least 0.5 gram of surfactant per litre in 0.1 molar aqueous sodium chloride solution, the ratio by weight of the alkyl orthoxylene sulphonate to the second surfactant lying in the range from 1:20 to 9:1.

2. A method according to claim 1 wherein the amount of alkyl orthoxylene sulphonate in the wash liquor is at least 0.05 g/l and the ionic strength in the wash liquor is not more than 0.05 mole/l.

3. A method according to claim 1 wherein a said second surfactant is present and the ionic strength in the wash liquor is at least 0.02 mole/l.

4. A detergent composition suitable for use in the method of claim 1, comprising:
from 1% to 50% by weight of a detergent active material which comprises an alkyl orthoxylene sulphonate; which composition:

i) does not contain more than 60% by weight of water-soluble alkali metal salt, or

ii) includes in the detergent active material an anionic or nonionic second surfactant which is sufficiently soluble as to be able to give a clear solution of at least 0.5 gram of surfactant per litre in 0.1 molar aqueous sodium chloride solution, the ratio by weight of the alkyl orthoxylene sulphonate to the second surfactant lying in the range from 1:20 to 9:1.

5. A detergent composition according to claim 4 which does not contain more than 60% by weight of water-soluble alkali metal salts and contains from 10 to 60% by weight of a water-insoluble detergency builder material.

6. A detergent composition according to claim 4 or claim 5 which does not contain more than 60% by weight of water-soluble alkali metal salts and the alkyl orthoxylene sulphonate constitutes at least 90% by weight of all the surfactant present.

7. A detergent composition according to claim 4, claim 5 or claim 6 which does not contain more than 60% by weight of water-soluble alkali metal salts and contains a nonionic surfactant with an HLB value below 10.5.

8. A detergent composition according to claim 4 which contains a said second surfactant and also contains from 15 to 95% by weight of water-soluble salts.

9. A detergent composition according to claim 4 or claim 8 wherein a said second surfactant is present in an amount less than the amount of alkyl orthoxylene sulphonate.

10. A detergent composition according to any one of claims 4 to 9 which contains at least one of:
fabric softening agents
fabric softening clay
lather booster
lather depressant
bleaching agent
fluorescent agent

organic polymer containing carboxylic or sulphonic acid groups
perfume, and
enzyme.