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LIQUID DETERGENT COMPOSITION

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The present invention relates to a pourable, concentrated liquid detergent composition which comprises an alkali metal higher alkyl aryl sulfonate detergent and a potassium polyphosphate in an aqueous alcoholic medium in homogeneous form, as hereinafter described and claimed.

In the detergent art, it has been common practice to prepare detergent compositions in powdered form, as obtained by spray-drying a slurry of the detergent composition for example. In these powdered products, alkaline builder salts such as sodium tripolyphosphate and pyrophosphate have been incorporated in significant amounts to improve the detergency or washing power for the laundering of soiled textile materials. Other salts such as sodium silicate, carbonate, carboxymethylcellulose and the like have been employed also in the preparation of powdered heavy-duty detergent compositions.

In accordance with the present invention, there are prepared heavy-duty liquid detergent compositions which consist essentially of about 5 to 25% by weight of alkali metal alkyl mononuclear aryl sulfonate detergent having an alkyl group of 8 to 15 carbon atoms and 10 to 50% by weight of a potassium polyphosphate selected from the group consisting of pyrophosphate and tripolyphosphate, the total content of organic detergent and potassium polyphosphate being a maximum of about 55% by weight, and an aqueous solubilizing medium in admixture therewith sufficient to form a liquid pourable in homogeneous form, said aqueous medium containing a saturated aliphatic monohydric alcohol of 2 to 3 carbon atoms in an amount from about 1/2 to 15% by weight.

A feature of the present invention relates to a liquid consisting essentially of the following ingredients proportioned within the stated ranges to form a pourable, substantially homogeneous aqueous liquid which consists essentially of about 5 to 20% by weight of said higher alkyl aryl sulfonate detergent, about 10 to 35% by weight of said potassium polyphosphate, the total amount of detergent and polyphosphate being at least about 20% by weight, about 4 to 12% by weight of an alkali metal lower alkyl aryl sulfonated hydrotropic salt, and preferably about 2 to 15% of a higher fatty acid alkylolamide, the total solids content being less than about 65% by weight, and a lower monohydric alcohol in an amount sufficient to obtain the desired physical properties thereof as further described herein. Various other significant features will be apparent in the following description.

The alkali metal alkyl mononuclear aryl sulfonate detergent having about 8 to 15 carbon atoms in the alkyl group which it is preferred to use is alkyl benzene sulfonate, though other similar detergents having a mononuclear aryl nucleus, such as alkyl toluene and alkyl xylene sulfonates, may be used. The alkyl substituent may be branched such as nonyl, dodecyl and pentadecyl groups including mixtures thereof which are derived from polymers of lower mono-olefins. The alkyl group may be straight-chained in structure such as the decyl, keryl and dodecyl groups. Preferred examples of this class are the alkyl benzene sulfonates having an average of about 12 to 15 carbons in the alkyl group. These sulfonate detergents are used in the form of their water-soluble alkali metal salts such as the sodium or potassium salts.

The potassium polyphosphate salts have the property of inhibiting precipitation of calcium and magnesium material

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in aqueous solution and of contributing to the heavy-duty performance of the liquid detergent product in washing operations. While they may be considered as derived from orthophosphoric acid or the like by the removal of molecularly bound water, any suitable means of manufacture may be employed. Such complex or molecularly dehydrated salts may be used in the form of the normal or completely neutralized salt such as pentapotassium triphosphosphate or the partially neutralized salt such as the potassium acid tripolyphosphate. It is preferred to employ pentapotassium tripolyphosphate and particularly tetrapotassium pyrophosphate, including any desired combination thereof.

The organic detergent and polyphosphate salts are employed in substantial amount such that the addition of a small portion of the liquid product to a washing bath will result in effective detergency and washing power. The proportions of these two ingredients are variable and should be suitably selected and proportioned so that there is formed a pourable, substantially homogeneous product by the solubilizing medium. As indicated the amount of detergent is selected from the range of 5 to 25% and usually up to 20% by weight. The amount of polyphosphate is within the range of 10 to 50% and usually up to 35% by weight with the preferred amount being about 15 to 30% of the composition. The total amount of organic detergent and polyphosphate should be a maximum of about 55% by weight. It is preferred that the total concentration be at least about 20% by weight. Amounts of detergent and polyphosphate totalling at least 20% by weight are incompatible in water resulting in a product which generally separates rapidly, usually within a few minutes. With proportions of detergent and phosphate within the stated ranges, pourable and substantially homogeneous liquids may be formed using an aqueous alcoholic solubilizing medium as described herein. As a general guiding principle, it is advisable to vary the detergent and polyphosphate contents conversely, i.e., relatively low amounts of one are used when relatively high amounts of the other are desired in the product.

The term "pourable" as used herein refers to the ability of the product to flow as a liquid from a container at normal room temperatures (e.g., about 20° C. to 25° C.). The liquid product may be of variable viscosity. It may be highly fluid and free-flowing, or fairly viscous when at rest but pourable freely from the container upon light shaking. The liquid may be packaged in any suitable container, however, such as metal, glass or plastic in the form of bottles, cans, drums, packets or bags. The term "homogeneous" as used herein means that the product has a satisfactory degree of stability during storage life against separation or formation of a plurality of distinct layers which either cannot be re-dispersed by light shaking or which separate rapidly after shaking. Such compositions include liquids which are clear, translucent or opaque depending upon the type and proportions of the various solids present therein. Any solid material present in the composition in excess of its solubility in the particular system is maintained in a non-caking suspension. It is characteristic that in such cases, upon light shaking, the product is readily converted to a substantially homogeneous liquid which is stable for a sufficient period of time, e.g., at least about 1 hour, so that each small portion added to a laundering bath or the like will have practically the same composition. The product exhibits a washing power which is comparable to the same quantity of commercial heavy-duty detergents in powdered form.

The aqueous alcoholic solubilizing medium should contain a saturated aliphatic monohydric alcohol of 2 to 3 carbon atoms such as ethanol, propanol or isopropanol so as to provide for desired physical properties of the liquid

composition. Depending upon the proportions of the other ingredients in a particular composition, the alcohol may serve a multiplicity of functions. Using selected proportions of detergent and phosphate within the ranges indicated, the alcohol in sufficient amount renders the detergent and phosphate more compatible in water. The appearance of the liquid such as a clear or opaque product is controllable by the proportions of detergent and phosphate employed in the aqueous medium using a sufficient amount of the alcohol. For example, in products containing about 10 to 20% phosphate and about 5 to 25% detergent where minimum amounts of both are employed or where the amounts vary conversely, the presence of a suitable amount of alcohol, e.g., usually about 10 to 15%, can provide for a clear liquid at room temperature. In other instances, such as where medium to high amounts of both detergent and phosphate are employed, the resulting products tend to be translucent to opaque with the alcohol preventing unduly rapid separation or formation of non-redispersible phases.

In addition, the alcohol permits desired modification of the viscosity, such as to make the product less viscous where high solids are employed and improve pourability thereby. It can provide for improved low temperature aging of the product such as the prevention of a precipitate at moderately low temperatures, e.g., about 40° F., which does not redisperse upon recovery to room temperature. The alcohol may adjust also the phase relationship so as to lower the cloud point and insure a clear liquid at a lower temperature if desired. In general, it tends also to minimize foaming and aeration of the product during manufacture and upon shaking of the container so as to permit pouring of uniform portions thereof. These improvements may be effected with relatively small amounts of alcohol, e.g., up to about 10% and usually up to 5% by weight, where the aqueous medium contains supplemental solubilizing agents such as the hydrotrope described herein to promote the stability of the system.

The amount of alcohol which should be employed varies with the particular formulation so as to achieve at least one of the above improvements in physical properties. In general, the alcohol is a solubilizing agent for said organic detergent in water but is relatively incompatible with inorganic salts. There is a critical maximum proportion of alcohol since an excessive amount will result in separation of the product into two or more layers which either separate rapidly or will not redisperse readily. The amount of alcohol used should not adversely affect the homogeneity of the liquid therefore, such as by separation of inorganic salt solution or by excessively thinning a high solids product where relatively high viscosity is desired as an aid in preventing rapid separation.

It is advantageous that the aqueous medium contain a water-soluble hydrotropic substance as a solubilizing agent in promoting the compatibility of the ingredients so as to form a homogeneous liquid product. The water-soluble organic sulfonated hydrotropes, particularly the alkali metal sulfonated salts having a lower alkyl group up to about 6 carbons, are effective in these products. It is preferred to employ an aryl sulfonate such as the sodium and potassium toluene and xylene sulfonate salts. Sulfonates made from xylene include orthoxylylene sulfonate, metaxylylene sulfonate, paraxylylene sulfonate and ethylbenzene sulfonate. Commercial xylene sulfonates usually contain metaxylylene sulfonate as the main ingredient. Analyses of typical commercial products show about 40-50% metaxylylene sulfonate, 10-35% orthoxylylene sulfonate and 15-30% paraxylylene sulfonate with 0-20% ethylbenzene sulfonate. Any suitable isomeric mixture may be employed, however. Suitable lower alkyl sulfate salts may be employed also, such as the alkali metal n-amyl and n-hexyl sulfates. The hydrotrope may be employed in variable amounts depending

upon the proportions of other ingredients but ordinarily it will be present in a suitable amount from about 4% to about 12%, preferably about 4 to 10%, by weight of the composition.

The presence of a combination of the hydrotropic sulfonate salt and the lower aliphatic alcohol permits increased flexibility for variation in formulation so as to obtain a range of physical properties which is not possible by the use of these ingredients individually. The hydrotropic salt may be employed in said range to form a clear or opaque liquid at room temperature. In certain formulations containing medium amounts of detergent and relatively low phosphate, it is possible to obtain a clear liquid at room temperature in the presence of sufficient hydrotropic salt in an aqueous medium. The presence of suitable amounts of alcohol in combination is advantageous for such physical characteristics as lowering of the cloud point, improvement in the low temperature aging of the product, etc., in addition to providing a co-solubilizing action with the hydrotrope permitting use of lesser amounts of the latter ingredient depending upon the particular formulation. In systems containing medium to high amounts of detergent and phosphate within the ranges described, there is produced a lotion-like or opaque liquid using the hydrotropic salt and alcohol in combination. There may be obtained homogeneous liquids with this combination using amounts of the alcohol and hydrotropic salt which individually do not provide a product of sufficient homogeneity or pourability. For example, where the solids concentration is at a high level, the amount of hydrotropic salt which may be added is limited since it will increase the solids content. Where the solids content is excessive, a non-pourable product will be obtained. The presence of the alcohol in combination will permit the formulation of the desired liquid product.

It is preferred that the liquid contain a higher fatty acid alkylolamide material in a sufficient amount to act as a suds builder during washing operations. In certain formulations, the alkylolamide provides an additional solubilizing effect. It should not be employed in an amount sufficient to destroy the desired physical properties since it is considered as part of the total solids content. In general, the alkylolamides may be employed in amounts of about 2 to 15% by weight in the product, and preferably from about 3 to 8%. The acyl radical of the alkylolamide is selected from the class of fatty acids having 8 to 18 carbons and each alkylol group has up to 3 carbon atoms usually. It is preferred to use the diethanolamides, isopropanolamides and monoethanolamides of fatty acids having about 10 to 14 carbons in the acyl radical. Examples are the lauric, capric, myristic and coconut diethanolamides, monoethanolamides and isopropanolamides, and mixtures thereof. There may be employed also the alkylolamides which are substituted by additional alkylol groups, suitable examples being the above monoethanolamides, diethanolamides and isopropanolamides condensed with one or two moles of ethylene oxide.

It has been found that pourable, homogeneous liquids may be prepared having a solids content of up to about 65% by weight in the aqueous alcoholic medium containing suitable proportions of said solubilizing agents. It is preferred to have a maximum of about 60% solids for opaque liquids and up to about 50% solids for clear solutions. The balance of the liquid content is primarily water since the liquid alcohol is used in small amounts. The amount of water will be suitably proportioned to provide sufficient solubilizing effects for the phosphates and similar inorganic salts so as to form a substantially homogeneous product. The amount of water is generally at least about 30% by weight and preferably from 40 to 60% of the liquid.

It has been found that these formulations are compatible with a suitable soil-suspending material which represents a further embodiment of the present invention. The addition of a soil-suspending material converts the sys-

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tem to a more complex one which has improved performance characteristics in washing operations. In general, the soil-suspending agent is a hydrophilic colloid which is dispersible in water and is maintained in suspension in the liquid product. It is preferred to use the alkali metal salt of a carboxy lower alkyl cellulose having up to 3 carbons in the alkyl group, such as the sodium and potassium salt of carboxymethylcellulose. The commercial grades of carboxymethylcellulose may be employed. Other known water-soluble cellulosic materials which may be used in compositions of the present invention are the lower alkyl and hydroxy alkyl ethers such as methylcellulose, ethylcellulose and hydroxyethylcellulose; and the various cellulose sulfate materials.

Other types of soil-suspending agents may be employed such as the water-soluble vinyl polymers. Examples are water-soluble polyvinyl alcohol which may contain minor amounts of polyvinyl acetate as commercially made. Water-soluble polyvinyl polymeric amides such as polyvinylpyrrolidone of suitable molecular weight may be employed also.

In general, the amount of the soil-suspending agent (including mixtures thereof) is usually from about 0.1 to about 2% by weight in the liquid product. It has been found that amounts within this range can be used in the preparation of substantially homogeneous products which exhibit a high level of soil suspension during washing. Various materials or mixtures may be employed which assist in maintaining the soil-suspending material in suspension or dispersion in the liquid product. Suitable mixtures of cellulosic compounds, or a mixture of a cellulosic compound with a vinyl polymer, or a mixture of a cellulosic compound with castor wax may be employed.

The liquid detergent product is prepared in any suitable manner. The hydrotropic salts, the polyphosphate and the organic detergent are added in the form of powders, aqueous solutions or slurries to the aqueous medium as desired. The alkylolamide is preferably added in molten or liquid form with agitation to form a homogeneous mixture. The alcohol may be added at any stage during the mixing operations as desired. The temperature of admixture is not critical and room temperatures or reasonable elevated temperatures may be employed.

The following examples are illustrative of the nature of the invention and it will be understood that the invention is not limited thereto. All parts are in approximate weight percent.

Example I

Ingredient:	Percent
Sodium dodecyl benzene sulfonate -----	12.00
Potassium tripolyphosphate -----	30.00
Potassium toluene sulfonate -----	8.00
Coconut diethanolamide -----	10.00
Sodium carboxymethylcellulose -----	1.80
Ethyl alcohol -----	7.00
Water -----	30.44
	99.24

the balance being minor amounts of perfume, coloring material, and optical bleach.

This product is prepared by the preferred method whereby the potassium tripolyphosphate is added slowly to the water, followed by introduction of the toluene sulfonate, the dodecyl benzene sulfonate, the coconut diethanolamide and the bleach and coloring material, each material being mixed thoroughly in the aqueous mixture before the next material is introduced. The perfume and ethanol are added followed by the slow addition of the carboxymethylcellulose with continued mixing until there is obtained a relatively thick gelatinous liquid. The mixture is transferred to a water-cooled colloid mill where it is homogenized for about 8 minutes. The pH is 9.3, specific gravity 1.3, and viscosity of about 1500 centipoises. The finished product has a final solvent analysis

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of 4-5% of ethyl alcohol and 27-30% water due to some loss of solvent during processing.

This product remains substantially homogeneous for many days and produces a high degree of washing power in both soft and hard water for laundering of soiled textile materials. The washing power corresponds to that of a commercially acceptable heavy-duty detergent composition in powder form.

Example II

The same formulation is used except that the coconut diethanolamide is reduced from 10% to 5% by weight, with a corresponding increase in the water content. This product is satisfactory also, and exhibits no apparent separation after about 8 hours.

Example III

A similar composition is formed by using the formulation of Example I but substituting an equivalent quantity of tetrapotassium pyrophosphate for the tripolyphosphate.

Example IV

A satisfactory composition is prepared according to the following formulation.

Ingredient:	Percent
Sodium dodecyl benzene sulfonate -----	12
Potassium tripolyphosphate -----	36
Potassium toluene sulfonate -----	6
Coconut diethanolamide -----	9
Sodium carboxymethylcellulose -----	0.8
Ethanol -----	6
Water (q.s. to 100%).	

Examples V-X

[Percent]

Ingredients	V	VI	VII	VIII	IX	X
Sodium tridecyl benzene sulfonate -----	5	5	25	5	20	25
Potassium pyrophosphate -----	10	20	10	50	35	15
Ethanol -----	14	11	12	14	15	10
Water (q.s. to 100%).						

In the above formulations, the sodium tridecyl benzene sulfonate is of about 90% purity, the balance being sodium sulfate formed in its manufacture. The alkyl group of the detergent is derived from a mixture of propylene polymers corresponding on the average to a tridecyl group.

All of the above products are pourable homogeneous liquids in use. The product of Examples V-VII are clear solutions at 77° F. The use of lesser amounts of alcohol results in a higher cloud point. The products of Examples VIII-X are opaque liquids which upon aging separate into two re-dispersible liquid phases and upon light shaking are converted to homogeneous products having satisfactory stability. As indicative of the adverse effect of excessive alcohol in a particular formulation, Example I modified to an ethanol content of 15% with a corresponding reduction in the water content results in a product which separates rapidly.

Example XI

Ingredients:	Percent
Sodium pentadecyl benzene sulfonate -----	20
Potassium tripolyphosphate -----	30
Potassium toluene sulfonate -----	4
Sodium carboxymethylcellulose -----	1.8
Ethanol -----	11
Water (q.s. to 100%).	

The above formulation is pourable as a homogeneous liquid upon light shaking due to some separation upon aging. The absence of alcohol and an increase of hydro-trope to 8% results in a non-pourable product. The use of insufficient alcohol, e.g., 7%, in the above formula

results in a liquid product which separates rapidly into two layers.

Example XXII

Ingredients:	Percent
Sodium tridecyl benzene sulfonate -----	19
Potassium pyrophosphate -----	15
Sodium xylene sulfonate -----	7
Coconut diethanolamide -----	3
Ethanol -----	4
Water (q.s. to 100%).	

The above formulation is a pourable, clear solution at room temperature whereas the omission of the alcohol results in a turbid liquid product.

Examples XIII-XV

[Percent]

Ingredients	XIII	XIV	XV
Potassium dodecyl benzene sulfonate -----	14.7	14	6.7
Tetrapotassium pyrophosphate -----	14.7	13	25.6
Sodium xylene sulfonate -----	11.8	7.5	11.4
Fatty acid alkylolamide ¹ -----	14.7	1.9	1.9
Ethanol -----	1.8	7.5	5.5
Water (q.s. to 100%).			

¹ The alkylolamide is a 1:1 mixture of isopropanolamides and diethanolamides having about a 70:30 ratio of lauric to myristic radicals.

The above formulations are clear solutions with low cloud points whereas in the absence of the alcohol the resulting products have two phases with cloud points above room temperature.

Examples XVI-XVII

[Percent]

Ingredients	XVI	XVII
Sodium dodecyl benzene sulfonate -----	12	8.6
Tetrapotassium pyrophosphate -----	20	20
Sodium xylene sulfonate -----	8	4
Coconut diethanolamide -----	2	
Coconut isopropanolamide -----		2.7
Ethanol -----	2	2-4
Sodium carboxymethylcellulose -----	0.5	0.5
Castor wax -----	0.3	0.3
Water (q.s. to 100%).		

The above formulations are pourable homogeneous liquids and exhibit improved physical properties after aging at 40° F. compared to products free of the alcohol.

Various other ingredients may be added or substituted if desired. In a liquid containing a plurality of components, the individual solubility characteristics of added materials may influence the physical properties of the liquid. It is understood therefore that such ingredients will be suitably selected and the proportions of the various components controlled as described herein so as to form a liquid product having the desired physical qualities. Thus, there may be included suitable amounts of various anionic sulfate or sulfonate or non-ionic organic detergents. Other adjuvant materials include compatible perfumes, coloring materials, corrosion inhibitors, anti-tarnishing agents, germicides, bleaching agents, optical bleaches or fluorescent dyes, and the like. If desired, there may be included other phosphate materials or similar builder salts such as silicates in the product provided that they do not substantially adversely affect the product.

This application is a continuation-in-part of my prior application Serial No. 532,735, filed September 6, 1955, and now abandoned.

Although the present invention has been described and illustrated with reference to specific examples, it is understood that modifications and variations of composition are contemplated within the scope of the appended claims.

What is desired to be claimed is:

1. An improved liquid detergent composition consisting essentially of about 5 to 25% by weight of alkali metal

alkyl mononuclear aryl sulfonate detergent having an alkyl group of 8 to 15 carbon atoms and 10 to 50% by weight of a potassium polyphosphate selected from the group consisting of pyrophosphate and tripolyphosphate, the total content of organic detergent and potassium polyphosphate being a maximum of about 55% by weight, and having a saturated aliphatic monohydric alcohol of 2 to 3 carbon atoms in an amount from about ½ to 15% by weight of the composition, the balance being substantially water in admixture therewith sufficient to form a liquid composition pourable in homogeneous form.

2. An improved liquid detergent composition consisting essentially of about 5 to 25% by weight of alkali metal alkyl benzene sulfonate detergent having an alkyl group of 8 to 15 carbon atoms and 10 to 50% by weight of a potassium polyphosphate selected from the group consisting of pyrophosphate and tripolyphosphate, the total content of organic detergent and potassium polyphosphate being within the range of about 20 to 55% by weight, and having ethanol in an amount from about ½ to 15% by weight of the composition, the balance being substantially water in admixture therewith sufficient to form a pourable, substantially homogeneous liquid composition.

3. A liquid detergent composition consisting essentially of about 5 to 20% by weight of alkali metal alkyl benzene sulfonate detergent having an alkyl group of 8 to 15 carbon atoms, about 10 to 35% by weight of potassium polyphosphate selected from the group consisting of pyrophosphate and tripolyphosphate, the total content of organic detergent and potassium polyphosphate being within the range of 20 to 55% by weight, about 4 to 12% by weight of an alkali metal organic sulfonated hydrotropic salt selected from the group consisting of alkali metal alkyl substituted benzene sulfonates having up to 2 carbon atoms in said alkyl substituent and alkyl sulfates having 5-6 carbon atoms, said aqueous alcoholic medium having about ½ to 15% by weight of a saturated aliphatic monohydric alcohol of 2 to 3 carbon atoms, the balance being substantially water in admixture therewith sufficient to form a pourable, substantially homogeneous liquid, the total solids content being less than about 65% by weight of the composition.

4. A liquid detergent composition as set forth in claim 3 which contains about 2 to 15% of a higher fatty acid alkylolamide having about 2 to 3 carbons in each alkylol group and about 8 to 18 carbons in the fatty acyl group.

5. A liquid detergent composition consisting essentially of the following ingredients proportioned within the stated ranges to form a pourable, substantially homogeneous product: about 5 to 20% by weight of alkali metal alkyl benzene sulfonate having an alkyl group of 8 to 15 carbon atoms, about 10 to 35% by weight of potassium polyphosphate selected from the group consisting of pyrophosphate and tripolyphosphate, the total content of organic detergent and potassium polyphosphate being within the range of 20 to 55% by weight, about 4 to 12% by weight of an alkali metal sulfonated hydrotropic salt selected from the group consisting of toluene and xylene sulfonates, about 2 to 15% by weight of a higher fatty acid alkylolamide having about 10 to 14 carbon atoms in the fatty acyl radical and 2 to 3 carbon atoms in the alkylol radical, about ½ to 15% by weight of a saturated aliphatic monohydric alcohol of 2 to 3 carbon atoms, and the balance being substantially water, the total solids content being less than 65% by weight of the composition.

6. A liquid detergent composition as set forth in claim 5 which contains about 0.1 to 2% by weight of hydrophilic colloid material selected from the group consisting of alkali metal carboxymethylcellulose, methylcellulose, ethylcellulose, hydroxyethylcellulose, polyvinylpyrrolidone and polyvinyl alcohol.

7. A liquid detergent composition consisting essentially of the following ingredients proportioned within the stated ranges to form a pourable, substantially homogeneous product: about 5 to 20% of alkali metal alkyl benzene

sulfonate having an alkyl group of about 12 to 15 carbon atoms, about 15 to 30% by weight of potassium polyphosphate selected from the group consisting of pyrophosphate and tripolyphosphate, about 4 to 12% by weight of an alkali metal sulfonated hydrotropic salt selected from the group consisting of toluene and xylene sulfonates, about 2 to 15% by weight of a higher fatty acid alkylolamide having about 10 to 14 carbon atoms in the fatty acyl radical and selected from the group consisting of diethanolamides, monoethanolamides and isopropanolamides, about ½ to 15% by weight of ethanol, and the balance being substantially water, the total solids content being less than 60% by weight of the composition.

8. A liquid detergent composition as set forth in claim 7 which contains about 0.1 to 2% by weight of sodium carboxymethylcellulose.

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