United States Patent Office 2,738,333 Patented Mar. 13, 1956

2,738,333

SURFACE-ACTIVE COMPOUNDS AND DETERGENT COMPOSITIONS CONTAINING SAME

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No Drawing. Application November 6, 1950, Serial No. 194,375

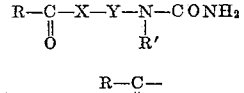
20 Claims. (Cl. 252—152)

The present invention relates to new urea type derivatives suitable in the preparation of surface-active compositions, and to synthetic detergent compositions containing the same having enhanced properties.

It has been accepted that surface-active compounds in general are characterized by the mutual presence in the molecule of both a hydrophobic or water-repellant group and a hydrophilic or water-attracting group. Many important variations in properties may be effected by varying the nature or type of such groups, and their relationship to each other in the molecule.

It is recognized in the art that many complex phenomenons are involved in the field of detergency, including such mechanisms as dispersion, interfacial tension, modification of micellar structure, etc. In addition, there are a multiple number of other variables involved in the determination of detersive power such as the nature of the essential detergent, the type of soil and substrate, the specific effect of various additives, etc. so as to render improvements of a detergent composition difficult and complex. In view of the varied nature of detergents, moreover, surface-active compounds designed for use in detergent compositions in general exhibit a certain degree of specificity of action.

In accordance with the present invention, a new class of compounds may be prepared which are useful in the production of surface-active compositions and the like. The novel compounds may be represented by the formula:

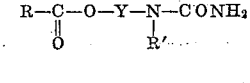


wherein



is a higher fatty acyl radical, X is a member selected from the group consisting of oxygen and imino radicals, Y is selected from the group consisting of alkylene and hydroxy alkylene radicals from 2 to about 6 carbon atoms, and R′ is selected from the group consisting of hydrogen, alkyl and hydroxyalkyl radicals. More particularly, it is preferred that R is a higher alkyl radical, X is selected from the group consisting of oxygen, and imino groups having a substituent selected from the group consisting of hydrogen, lower alkyl and lower hydroxyalkyl groups, and R′ may be similarly hydrogen, lower alkyl, and lower hydroxyalkyl. Where hydroxy groups are indicated, either mono- or polyhydroxy groups are included therein. The lower alkyl and hydroxyalkyl groups will usually be up to about five carbons, and preferably about 2–3 carbon atoms, and Y may either be straight or branched-chain in structure, preferably about 2–4 carbon atoms.

Included within the ambit of the invention are the following preferred classes of compounds which may be represented by the following structural formulas:

(1) 

wherein



is an acyl radical derived from a higher fatty acid of about 8 to about 22 carbon atoms, R being preferably a higher alkyl group of about 10 to about 18 carbons, Y is selected from the group consisting of alkylene and hydroxy alkylene radicals of 2 to about 6 carbon atoms and preferably 2–4 carbons, and R′ is selected from the group consisting of hydrogen, lower alkyl and lower hydroxy alkyl radicals. Suitable examples of such compounds wherein R′ is hydrogen are: lauric acid ester of hydroxyethyl urea, myristic acid ester of hydroxyethyl urea, lauric acid ester of hydroxypropyl urea, lauric acid ester of hydroxybutyl urea, mixed coconut oil fatty acid ester of hydroxyethyl urea, lauric acid ester of hydroxyisopropyl urea, etc. Examples wherein R′ is alkyl are lauric acid ester of N-hydroxyethyl, N-ethyl urea, myristic acid ester of N-hydroxyethyl, N-methyl urea, etc. Examples wherein R′ is a hydroxyalkyl group are monolauric acid ester of N,N di-hydroxyethyl urea, monomyristic acid ester of N,N di-hydroxypropylurea, monolauric acid ester of N,N di-hydroxyisopropylurea, etc.

These compounds may be prepared in any suitable manner. A convenient mode of synthesis comprises esterifying the hydroxyl group of an appropriate alkylolamine with a higher fatty acylating compound to form the corresponding ester linkage and reacting the amino group with a cyanate to form a urea linkage therewith. These reactions may be conducted in any suitable order. Thus, the alkylolamine may be reacted with the cyanate to form the unsymmetrical hydroxyalkyl urea, which may thereafter be esterified with the suitable higher fatty acylating compound to form the desired ester. Alternatively, the alkylolamine may be condensed with the higher fatty acid and the like to form the ester, which may be reacted with the cyanate compound to form the desired urea type linkage.

These higher fatty acid acylating substances are well known in the art and include for example the higher fatty acid per se, the acid halide, acid anhydride, ester, etc. These higher fatty acylating substances may be derived from pure, impure or commercial grades of higher fatty acids and the like. More particularly, they may be derived from coconut oil, palm oil, tallow, and other fatty oils, fats, greases of animal, vegetable or marine origin. Alternatively, they may be of synthetic origin such as the product of oxidation of hydrocarbons, etc. Further suitable examples of these acylating compounds are lauric, stearic, myristic, oleic, palmitic acids and acid chlorides, etc., and mixtures thereof such as coconut oil and palm oil fatty acids.

The suitable alkylolamines are the primary and secondary alkylolamines having an alkylol group of two to about six and preferably about two to four carbons. It is preferred to use the primary alkylolamines such as monoethanolamine, monoisopropanolamine, monopropanolamine, monobutanolamine, 2-amino 2-methyl propanol, trimethylolaminomethane, etc. Suitable secondary alkylolamines are ethylethanolamine, methylethanolamine, diethanolamine, diisopropanolamine, diglyceryl-amine, etc.

Any suitable cyanate compound may be utilized as a reactant, preferably the water-soluble inorganic salts, such as the alkali metal salts, e. g. sodium and potassium cyanate.

Illustrative of the unitary process of preparing the higher fatty acid esters of the indicated urea derivatives is the initial preparation of the urea reactants followed by esterification. The hydroxyalkyl urea intermediates are derived by the reaction, in an aqueous or alcoholic

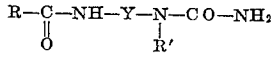
medium, of the indicated alkylolamine in the form of its salt with a suitable water-soluble acid (e. g. hydrochloric, acetic, etc.) with a suitable cyanate compound. An addition complex forms which rearranges to produce the desired urea linkage. The reaction may be represented graphically as follows using monoethanolamine and potassium cyanate:

$$HOCH_2—CH_2—NH_2 \cdot HCl + KOCN \rightarrow HOCH_2—CH_2—NHCONH_2 + KCl$$

Any suitable proportions of reactions may be utilized, however it is preferred to employ stoichiometric proportions of amine, acid and at least a stoichiometric amount of cyanate compound. The reaction may be conducted at low temperatures, such as room temperature for example, but preferably at slightly elevated temperatures such as up to refluxing temperatures.

The desired higher fatty ester derivative may be produced by the condensation of the acylating substances with these hydroxyalkylurea compounds. Any suitable proportions of each reactant may be utilized although it is preferred that at least equivalent amounts and preferably an excess of the urea derivative be employed. Any suitable condensation temperatures may be employed. Using fatty acids or anhydrides illustratively as reactants it is preferred that condensation temperatures be sufficient to distill or otherwise remove the desired amount of water formed during the reaction. It is preferred to use the fatty acid halides (e. g. acid chlorides) as reactants, generally at low temperatures, such as below about 100° C. and preferably not substantially above about 50° C., in an aqueous or organic solvent medium. In the latter procedure it is generally desired to have present an alkaline neutralizing agent such as sodium hydroxide, sodium bicarbonate etc. to neutralize the hydrochloric acid, for example, formed during the condensation reaction. Thereafter the product may be further purified in any suitable manner if desired.

(2)



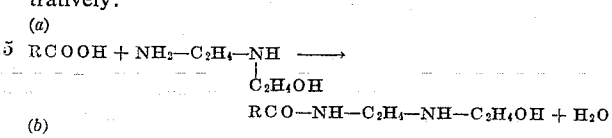
wherein R and Y have the significance indicated with compounds of group (1) supra, and R′ is a lower alkylol group. This lower alkylol may be mono- or polyhydroxy, usually up to about five carbons, and preferably about 2–3 carbons.

Suitable examples of such compounds are lauric amide of N-aminoethyl, N-hydroxyethyl urea; myristic amide of N-aminoethyl, N-hydroxyethyl urea; lauric amide of N-aminoisopropyl, N-hydroxyethyl urea, etc.

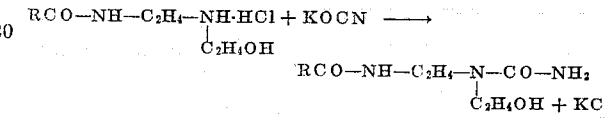
These compounds may similarly be prepared in any suitable manner. A preferred method comprises condensing the primary amino group of an N-alkylol aliphatic diamine with a higher fatty acylating compound to form the corresponding amide linkage, and reacting the residual amino group with a cyanate compound to form a urea linkage therewith. These reactions may be conducted in any suitable order, consideration being given by those skilled in the art to the reactivity of the functional groups. The suitable higher fatty acid acylating substances and cyanates set forth supra are equally applicable herein. Any suitable N-alkylol aliphatic diamine may be employed for the reactions such as N-hydroxyethyl ethylene diamine, N-hydroxyethylpropylene diamine, etc. The condensation reaction to produce the amido linkage may be effected under conditions substantially the same as those required for esterification in the preparation of the compounds of group I. The reaction may be conducted by mixing suitable proportions of the initial reactants in an aqueous or mutual solvent (e. g. acetone medium). In general, the reaction may be conducted by employing substantially stoichiometric ratios of the reactants as indicated by the typical equations set forth below. An excess of the amino compound over the acylating material is preferably used to obtain the desired monoamides and to prevent the formation of multi-acylated

derivatives such as polyamides and amide esters which would reduce the yield of the desired product. Where acid chlorides are used as the acylating material, a neutral or alkaline medium may be maintained by the presence during the reaction of a suitable alkaline neutralizing agent to neutralize liberated acid. The reaction of the resulting product containing a reactive hydrogen atom on an amino nitrogen with cyanate to form the urea linkage is substantially the same as indicated above and is incorporated herein by reference. The reaction may be represented as follows using hydroxy ethylene diamine, hydrochloric acid and potassium cyanate illustratively:

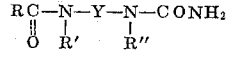
(a)



(b)



(3)



wherein R and Y have the significance indicated above, and R′ and R″ may be the same or different, each being selected from the group consisting of hydrogen and lower alkyl. Suitable examples are lauric acid amide of N-aminoethyl urea, caprylic acid amide of N-aminopropyl urea, myristic acid amide of N–3 amino, 2 hydroxy propyl urea, mixed coconut fatty acid amides of N-aminoethyl, N-ethyl urea, etc.

These compounds may be prepared similarly to the compounds of groups (1) and (2). The higher fatty acylating substances, cyanates, conditions of reactions, etc. are all equally applicable and incorporated by reference herein. Suitable amine reactants are ethylene diamine, 1,3 propylene diamine, hexamethylene diamine, 1,3 diaminopropanol, 1,3 diaminoisopropanol, N-ethyl ethylene diamine, etc. The type reactions are conveniently illustrated in Equations (*a*) and (*b*) set forth in group II with the necessary modification that the amine reactants of group III do not possess the N-alkylol group characteristic of the compounds of group II.

The following examples are illustrative of the preparation of these compositions and it will be understood that the invention is not limited thereto:

EXAMPLE I

1 mole of ethanolamine is dissolved in water, and neutralized to pH 4 with 1 mole of aqueous hydrochloric acid. A separately made solution of 1–1.1 moles of potassium cyanate in water is added with stirring, and the resulting solution evaporated to dryness. The residue is taken up in 95% ethanol, and filtered to remove precipitated salts. The filtrate is evaporated once again, and is reacted with 1 mole of lauroyl chloride in an acetone medium at 20° C. in the presence of 1 mole of dry sodium bicarbonate to take up the HCl formed. The reaction product is filtered free of precipitated sodium chloride, the filtrate evaporated, and the residue recrystallized from benzene, resulting in large, white, fatty scales, melting at 78° C. The desired product has the formula:

$$C_{11}H_{23}COOC_2H_4—NH—CO—NH_2$$

The product foams readily and exhibits good surface-active properties.

EXAMPLE II

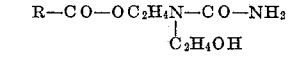
The process of Example I is repeated using monopropanolamine, sodium cyanate, and myristoyl chloride as the suitable reactants, resulting in the desired reaction product having the formula:

$$C_{13}H_{27}COOC_3H_7—NH—CO—NH_2$$

This product behaves similarly in solution as the reaction product of Example I.

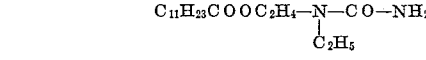
EXAMPLE III

The process of Example I is repeated using coconut fatty acid chlorides and diethanolamine as reactants, forming a final desired condensation product having the formula:



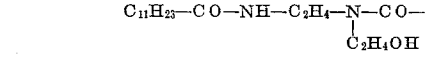
EXAMPLE IV

The process of Example I is repeated using lauric acid chloride and N-ethyl ethanolamine as reactants, forming a final desired condensation product having the formula:



EXAMPLE V

1 mole of lauric acid and 1–1.1 moles of amino ethyl ethanolamine (also called hydroxyethyl ethylene diamine) are heated together to 155–165°, until about 1 mole of water has been distilled off. Any excess amine present is then removed by applying reduced pressure or vacuum. The residue, mainly the fatty acid monoamide of amino ethyl ethanolamine, is dispersed in a suitable amount of water and solubilized by neutralizing to a pH of 3–5 with 1 mole of aqueous hydrochloric acid. A solution of 1–1.1 mole of potassium or sodium cyanate in water is added and the solutions suitably mixed and heated on a steam bath. After 1–2 hours of heating, the supernatant oily product is freed from the aqueous salt solution, and the oily phase is suitably dehydrated, e. g. by adding alcohol and distilling off alcohol-water, or azeotropically with a solvent such as benzene. (However, the oily phase may be used as is without further purification.) When the solvents are removed, a water soluble product is obtained. The product foams in aqueous solutions, and exhibits surface-active properties. The desired product has the formula:

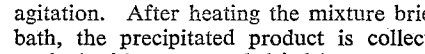


EXAMPLE VI

The procedure of Example V is repeated using mixed coconut fatty acid chlorides as a reactant with aminoethyl ethanolamine at a temperature of about 20–40° C. in the presence of about a mole of sodium hydroxide as neutralizing agent.

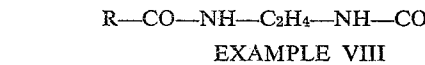
EXAMPLE VII

1 mole of a fatty acid monoamide of ethylene diamine is prepared by reacting coconut fatty acids with a large excess of ethylene diamine at about 150° C., distilling excess amine off in vacuo at a low temperature, and isolating the amide by precipitation with CO_2 from alcoholic solution. The reaction product is dissolved in water by neutralizing to a pH 3–5 with 1 mole of aqueous hydrochloric acid, and a solution of 1–1.1 mole of potassium or sodium cyanate in water is added slowly with vigorous agitation. After heating the mixture briefly on a steam-bath, the precipitated product is collected on a filter, washed with water, and dried in a stream of air. It is a solid of high melting point which is relatively insoluble in water, poorly soluble in alcohol and other organic solvents. The coconut derivative thus made melted at 144–5° C. and can be recrystallized from 80% alcohol yielding white scales, melting at 150–1° C. The desired product has the formula:



EXAMPLE VIII

The procedure of Example VII is repeated using lauric acid, 1,2 propylene diamine and potassium cyanate as the reactants, forming a final desired condensation product having the formula:



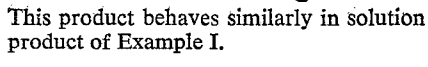
EXAMPLE IX

The procedure of Example VII is followed substituting 1,3 diamine propanol as the suitable amine reactant. The desired condensation product has the formula:



EXAMPLE X

The procedure of Example VII is repeated using N-ethyl ethylene diamine as the amine reactant. The desired product has the formula:



As indicated, these essentially non-ionic surface-active compounds may be used in the formation of detergent compositions. More particularly, they may be used in combination with other surface-active materials such as the synthetic anionic sulfated or sulfonated organic detersive compounds, in view of the fact that they are in general, fairly compatible therewith.

It is a significant feature of the present invention that the above complex urea derivatives may be incorporated in minor proportions in detergent compositions consisting essentially of anionic detergents to achieve a significant enhancement in surface-active properties such as detergency or sudsing primarily. Optimum results in enhancement of detergency appear to be achieved with the compounds of group I and II described above.

These novel detergent compositions of the present invention contain as the active ingredient the anionic sulfated and sulfonated detergents, including suitable mixtures thereof. Included therein are the aliphatic sulfated or sulfonated agents, such as the aliphatic acyl-containing compounds wherein the acyl radical has about 8 to about 22 carbon atoms, and, more particularly, the aliphatic carboxylic ester type, containing at least about 10 and preferably about 12 to about 26 carbon atoms to the molecule. Among the aliphatic detersive compounds, it is preferred to use the sulfated aliphatic compounds having about 12 to about 22 carbon atoms. As suitable examples of aliphatic detergents may be found the sulfuric acid esters of polyhydric alcohols incompletely esterified with higher fatty acids, e. g. coconut oil monoglyceride monosulfate, tallow di-glyceride monosulfate; the long chain pure or mixed higher alkyl sulfates, e. g. lauryl sulfate, cetyl sulfate, higher fatty alcohol sulfates derived from coconut oil; the hydroxy sulfonated higher fatty acid esters, e. g. higher fatty acid esters of 2,3 di-hydroxy propane sulfonic acid; the higher fatty acid esters of low molecular weight alkylol sulfonic acids, e. g. oleic ester of isethionic acid; the higher fatty acid ethanolamide sulfates; the higher fatty acid amides of amino alkyl sulfonic acids, e. g. lauric amide of taurine, and the like.

It is a feature of this invention that the effects are particularly enhanced with the alkyl aryl sulfonate detergents. These aromatic sulfonate detergents are also known in the art. They may be mononuclear or polynuclear in structure. More particularly, the aromatic nucleus may be derived from benzene, toluene, xylene, phenol, cresols, naphthalene, etc. The alkyl substituent on the aromatic nucleus may vary widely, as long as the desired detergent power of the active ingredient is preserved. While the number of sulfonic acid groups present on the nucleus may vary it is usual to have one such group present in order to preserve as much as possible a balance between the hydrophilic and hydrophobic portions of the molecule.

More specific examples of suitable alkyl aromatic sulfonate detergents are the higher alkyl aromatic sul-

fonates. The higher alkyl substituent may be branched or straight-chain in structure, and comprise decyl, dodecyl, keryl, mixed long-chain alkyls from polymeric lower mono-olefins, etc. Preferred examples of this class are the higher alkyl mononuclear aryl sulfonates wherein the alkyl group is about 8 to about 22, and preferably about 12 to about 18 carbon atoms. More particularly it is preferred to use the higher alkyl benzene sulfonates wherein the higher alkyl group averages about 12 to about 16 carbon atoms. For example, propylene may be polymerized to the tetramer and condensed with benzene in the presence of a Friedel-Crafts catalyst to yield essentially the dodecyl benzene derivative which is suitable for sulfonation to the desired sulfonate compounds.

These various anionic detergents are generally used in the form of their water-soluble salts, such as the alkali metal, alkaline earth metal, ammonium, amine, and alkylolamine salts. While the sodium, potassium, ammonium and alkylolamine (e. g. mono-, di-, and triethanolamine) salts are preferred ordinarily, other salts such as the lithium, calcium, and magnesium salts may be used if desired. For general use, it is ordinarily preferred to use the sodium and potassium salts. For certain specialized uses, it may be preferred to selected the ammonium and alkylolamine salts in view of their generally greater solubility in aqueous solution. The concentration of these water-soluble salts (including suitable mixtures thereof) in the detergent compositions of the present invention is generally at least about 10% by weight of total solids, and preferably from about 10–50%. With built compositions, particularly in particulate form, an active ingredient content of 10–50%, and preferably about 15–40% yields highly satisfactory results. Compositions with very high concentrations of these active ingredients are prepared for specialized uses generally.

The amount of these additives in the detergent composition is less than the weight of the active ingredient and is generally minor in proportion thereto and effective to produce the desired improvements, particularly in detersive capacity. Generally, the proportion of additives should be from about 5 to about 50% of the weight of the active ingredient and will prefrably be from about 1 to about 15% by weight of the total detergent composition. Particularly effective results have been achieved where the additives are utilized in amounts within the range of about 1 to about 10% of the total composition.

The additives may be incorporated with the active ingredient at any point during the manufacturing process at which subsequent operations will not adversely modify the properties of the detergent compositions. A variety of procedures which have proved to be convenient, economical, and productive of best results are: the additives may be added to a hot aqueous slurry of about 40 to 50% concentration of the active ingredient with vigorous stirring to form a smooth, uniform and homogeneous paste, the additives may be dissolved in a suitable solvent and added to the slurry of the active ingredient, or a mixture or emulsion of the additives in water with a minor proportion of the active ingredient may be incorporated into the slurry.

Thereafter, these compositions may be prepared in the form of solutions, pastes or as dry or partially hydrated solid products, preferably in a finely divided condition. It is preferred to prepare the products in particulate form. Accordingly, the slurry of the detergent composition may be subjected to any suitable drying operations and converted to particle form. The mixture may thus be subjected to conventional spray-drying, roll drying or drum drying operations utilizing temperatures above about 212° F. to obtain homogeneous detersive particles.

It is common to employ various adjuvant materials in synthetic detergent compositions. The detergent compositions of the present invention may include any of these substances employed by the art in admixture with such detergent compositions generally, provided the use of any such materials does not completely neutralize or remove the effect of the additives in the relationship set forth. These adjuvant builders or additives may be inorganic or organic in structure and may be mixed with the active ingredient in any suitable manner. Such convenient inorganic builders or additives as the various alkali metal phosphates, e. g. tripolyphosphate, hexametaphosphate, tetrapyrophosphate) the alkali metal silicates, sulfates, carbonate, etc. may be employed in these compositions. Suitable organic materials such as sodium carboxymethylcellulose may also be employed herein. It is preferred that the detersive compositions in particulate form contain major amounts of alkaline builders, particularly the inorganic water soluble phosphates. The total amount of phosphate compounds should be a minimum of at least about 10%, and preferably from about 10 to about 60% by the weight of the detergent composition for best results. An amount of active ingredient of about 15 to 50% with the requisite minor proportion of additives and about 20 to about 60% total phosphate compounds exhibits particularly desirable properties for a detergent composition.

The additives exert their primary detersive effects on an activation of the soil removal power. Such results may be illustrated by the application of soil removal tests, using a testing procedure which involves the uniform soiling, washing with particular detergent compositions at 110° F. +2° F., and drying a large number of cotton swatches. The whiteness of the various test swatches are measured by a Hunter reflectometer. The units of soil removed may be calculated by subtracting the average reflectivity of unwashed control samples from the washed swatches.

Table I indicates the percent change in soil removal on soiled cotton swatches using a standard detergent composition consisting essentially of 20% sodium salts of dodecyl benzene sulfonate (the dodecyl group being derived from a propylene tetramer,) 40% sodium tripolyphosphate, 3% sodium carbonate and 37% sodium sulfate at 0.4% concentration in soft tap water, with and without additive. The percentages listed in the table for each concentration of additive represent the present change in soil removal using as a standard the above detergent composition without organic additive. A (+) value indicates improved soil removal.

Table I

Additive	Percent	Percent Change in Soil Removal on Cotton Soil
Lauric acid ester of N-hydroxyethyl urea	1	+4
Do	2	+8
Do	3	+16
Do	4	+25
Do	5	+39
Do	7	+40
Do	10	+41
N-Lauramidoethyl N-Hydroxyethyl urea	2	+6
Do	5	+15
Do	10	+44
Coconut fatty acid amides of N-aminoethyl urea	2	+4
Do	5	+4
Do	10	+13

In each particular detergent composition there is an optimum proportion or concentration which may be determined by routine tests.

In general, the compounds represented by groups 1 and 2 supra exert an even more remarkable and significant improvement in soil removal on woolen fabrics, in comparison to the desirable effects achieved on cotton soil. Using substantially the same testing conditions at 90° F., the percent change in soil removal in wool soil is set forth in Table II.

Table II

Additive	Percent	Percent Change in Soil Removal on Woolen Soil
Lauric acid ester of N-hydroxyethyl urea	1	+10
Do	2	+17
Do	3	+37
Do	4	+52
Do	5	+71
Do	7	+88
Do	10	+107
N-lauramidoethyl N-hydroxyethyl urea	2	+10
Do	5	+41
Do	10	+81

The marked improvement resulting from the use of these additives on wool soil is graphically evident from the data.

Another important feature is the effect of these additives on the foaming characteristics of the detergent composition. The effects on the foaming characteristics can be studied quantitatively for a given composition. The pour foam test designed for the comparative study of the relative foam stability of liquids and appropriate apparatus for carrying out the test is set forth in U. S. Patent Number 2,315,983 to Ross and Miles. The foam height in millimeters of the solution tested in accordance with this patent is read at various time intervals and is an indication of foam stability.

Using the compositions disclosed in Table II with varying amounts of additives in 0.25% concentration in distilled and in hard water, it may be observed that these additives have no deleterious action on the initial volume or stability of foam of the composition. Such results are particularly important since it is desirable that in general the detergent compositions exhibit excellent foaming properties, particularly for consumer appeal and certain home and industrial uses. Many of the selected known organic additives have a pronounced adverse effect on foaming properties particularly foam stability, whereas these novel compositions exhibit marked foam stability in the relationship set forth.

In the same relationship the cocoylamidoethyl urea additives and the like appear to act as an inhibitor on the foam stability. Such effects would be of advantage in certain applications of detergent compositions where marked foaming properties are relatively unimportant or even undesirable. Thus, in detergent compositions designed for machine washing under certain conditions it may be desirable to have a minimum of foaming power.

Moreover, it has been found that in general these compositions tend to increase the tolerance of such detergent compositions in the assimilation or holding in suspension of the maximum amount of dirt, grease etc. with less foam loss than is found without the use of these additives.

Such results may be evidenced by soil tolerance tests, which are essentially the pour foam tests modified by the addition of a small amount of a soil to the detergent solution before testing. The results in such tests using 0.25% concentration in distilled water of the basic detergent compositions used in Tables I and II and containing varying amounts of additive are set forth in Table III.

Table III

Additive	Percent	0 min.	5 min.	10 min.	15 min.
None		195	170		
Lauric acid ester of N-hydroxyethyl urea	3	210	210	205	200
	5	210	210	210	210
	7	210	210	210	205
N- lauramidoethyl- N- hydroxyethyl urea	3	230	230	230	225
	5	230	230	225	220
	7	230	230	230	230
Coconut fatty acid amides of N-aminoethyl urea	1	220	215	70	50

The following examples are additionally illustrative of

the nature of the present invention and it will be understood that the invention is not limited thereto:

EXAMPLE XI

A detergent composition is prepared by forming about a 60% solids slurry containing on a solids basis about 35% sodium propylene tetramer benzene sulfonate salt, 40% sodium tripolyphosphate, 15% sodium sulfate, 5% lauric acid ester of N-hydroxyethyl urea and the remainder minor amounts of sodium chloride, sodium hydroxide, sodium carboxymethylcellulose, etc. This slurry is agitated at about 140° F. in a conventional soap crutcher to form a homogeneous mixture. The slurry is submitted to spray-drying with heated air at a temperature of about 350° F. with a resultant moisture loss of about 40%. The resulting composition is recovered in the form of beads, and possesses a high degree of detersive and foaming properties in both hard and soft water.

EXAMPLE XII

The procedure of Example XI is repeated with the modifications that the active ingredient consists essentially of the keryl benzene sulfonate salt, the organic additive is the N-lauramidoethyl, N-hydroxyethyl urea and the slurry is roll dried at about 50 lbs. steam pressure to flake form. This composition also possesses highly desirable detersive properties.

EXAMPLE XIII

Using the procedure of Example XI an improved detergent composition is prepared from the following components: 20% sodium lauryl sulfate, 40% sodium tripolyphosphate, 35% sodium sulfate, and 5% myristic acid ester of N-hydroxyethyl urea.

EXAMPLE XIV

Another suitable detergent composition is prepared by compounding 25% sodium coconut monoglyceride sulfate, 10% diacid disodium pyrophosphate, 62% sodium sulfate and 3% lauric acid ester of N-hydroxyethyl urea.

Other formulations productive of desired results are:

EXAMPLE XV

	Percent
Sodium coconut alcohol sulfates	16
Sodium chloride	1
Sodium tripolyphosphate	38
Tetrasodiumpyrophosphate	15
Sodium sulfate	25
Lauric acid ester of N-hydroxyethyl urea	5

EXAMPLE XVI

	Per cent
Sodium dodecylbenzene sulfonate	9
Sodium lauryl sulfate	10
N-lauramidoethyl N-hydroxyethyl urea	4
Sodium carboxymethylcellulose	0.5
Sodium carbonate	1.5
Sodium sulfate	20
Sodium tripolyphosphate	55

EXAMPLE XVII

	Per cent
Higher fatty acid amides of taurine derived from coconut oil	25
Sodium sulfate	30
Sodium tripolyphosphate	40
Lauric acid ester of N-hydroxyethyl urea	5

Certain general conclusions are apparent from the many tests which have been conducted to determine the effectiveness of the substituted urea additives in the relationship set forth. The most appropriate additive and its most effective concentration for each particular sulphonated or sulphated detergent composition may be suitably determined by routine controls. In each case the intended use (e. g. hard or soft water, rug shampoos or machine washing compositions, etc.) and the proper wash-

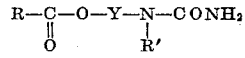
ing conditions should be taken into consideration in order to derive the maximum beneficial effects.

The term "consisting essentially of" as used in the definition of the ingredients present in the composition claimed is intended to exclude the presence of other materials in such amounts as to interfere substantially with the properties and characteristics possessed by the composition set forth but to permit the presence of other materials in such amounts as not substantially to affect said properties and characteristics adversely.

Although the present invention has been described with reference to particular embodiments and examples, it will be apparent to those skilled in the art that variations and modifications of this invention can be made and that equivalents can be substituted therefor without departing from the principles and true spirit of the invention.

Having described the invention what is desired to be secured by Letters Patent is:

1. A new surface-active urea derivative characterized by the formula:

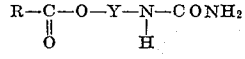


wherein



is a higher fatty acyl radical, Y is selected from the group consisting of alkylene and hydroxyalkylene radicals of 2–6 carbon atoms, and R′ is selected from the group consisting of hydrogen, lower alkyl and lower hydroxyalkyl radicals.

2. A new surface-active urea derivative characterized by the formula:

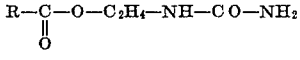


wherein



is a higher fatty acyl radical, and Y is an alkylene radical of 2–6 carbon atoms.

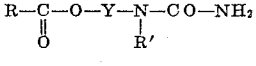
3. A new surface-active urea derivative characterized by the formula:



wherein R is an alkyl group of about 10 to about 18 carbon atoms.

4. As a new chemical compound, lauric acid ester of N-hydroxyethyl urea.

5. A new surface-active urea compound characterized by the formula:

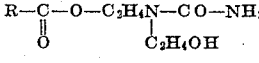


wherein



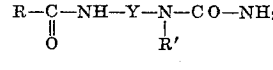
is a higher fatty acyl radical, Y is an alkylene radical of 2–6 carbon atoms and R′ is a lower hydroxyalkyl radical.

6. A new surface-active urea derivative characterized by the formula:



wherein R is a higher alkyl group of about 10 to 18 carbon atoms.

7. A new surface-active urea derivative characterized by the formula:

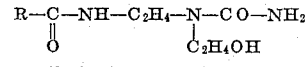


wherein



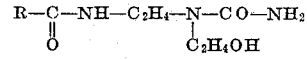
is a higher fatty acyl radical, Y is selected from the group consisting of alkylene and hydroxyalkylene radicals of 2 to 6 carbon atoms, and R′ is a lower alkylol group.

8. A urea derivative characterized by the formula:



wherein R is an alkyl group of about 10 to about 18 carbon atoms.

9. As a new surface-active urea derivative, N-lauramidoethyl N-hydroxyethyl urea having the formula:

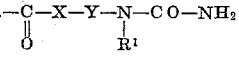


wherein



is lauroyl.

10. A detergent composition consisting essentially of a detergent selected from the class consisting of the water-soluble anionic sulfate and sulfonate detergents, and a compound represented by the formula:

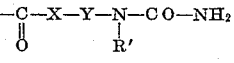


wherein



is a higher fatty acyl radical, X is a member of the class consisting of oxygen and imino groups, Y is selected from the class consisting of alkylene and hydroxyalkylene radicals of 2 to about 6 carbon atoms, and R′ is selected from the class consisting of hydrogen, lower alkyl and lower hydroxyalkyl groups, the amount of said compound being less than the weight of said detergent and effective in combination therewith to increase the detersive power thereof in aqueous solution.

11. A detergent composition consisting essentially of a detergent selected from the class consisting of the water-soluble anionic sulfate and sulfonate detergents, and from about 1 to 15% by weight of a compound represented by the formula:

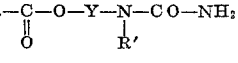


wherein



is a saturated higher fatty acyl radical, X is a member of the class consisting of oxygen and imino groups, Y is selected from the class consisting of alkylene and hydroxyalkylene radicals of 2 to about 6 carbon atoms, and R′ is selected from the class consisting of hydrogen, lower alkyl and lower hydroxyalkyl groups.

12. A detergent composition consisting essentially of a detergent selected from the class consisting of the water-soluble anionic sulfate and sulfonate detergents, and a minor proportion of a urea derivative represented by the formula:



wherein



is a higher fatty acyl radical, Y is selected from the class consisting of alkylene and hydroxyalkylene radicals of 2 to about 6 carbons, and R′ is selected from the class consisting of hydrogen, lower alkyl and lower hydroxyalkyl

radicals in an amount sufficient to improve the soil removal power thereof.

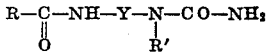
13. A detergent composition consisting essentially of a water-soluble detergent selected from the group consisting of the anionic sulfates and sulfonates, and a higher fatty acid ester of a N-hydroxyalkyl urea, said hydroxyalkyl group having from about 2 to about 6 carbon atoms, the amount of said urea compound being minor in proportion to said detergent and effective in combination therewith to produce enhanced detersive power thereof in aqueous solution.

14. An improved detergent composition consisting essentially of a higher alkyl aryl sulfonate detergent, and a higher fatty acid ester of a N-hydroxyalkyl urea, said hydroxyalkyl group having from about 2 to about 6 carbon atoms, the amount of said urea compound being minor in proportion to said detergent and effective in combination therewith to produce enhanced detersive power thereof in aqueous solution.

15. A detergent composition consisting essentially of a water-soluble higher alkyl aryl sulfonate detergent, and a higher fatty acid ester of a N-hydroxyalkyl urea, said hydroxyalkyl group having from about 2 to about 4 carbon atoms, the amount of said urea compound being minor in proportion to said detergent and effective in combination therewith to produce enhanced detersive power thereof in aqueous solution.

16. A detergent composition consisting essentially of a detergent selected from the group consisting of water-soluble organic sulfate and sulfonate detergent, and lauric acid ester of N-hydroxyethyl urea in an amount sufficient to improve the soil removal power thereof.

17. A detergent composition consisting essentially of a detergent selected from the class consisting of the water-soluble anionic sulfate and sulfonate detergents, and a minor proportion of a compound having the formula:

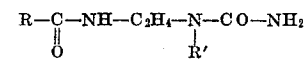


wherein



is a higher fatty acyl radical, Y is selected from the class consisting of alkylene and hydroxyalkylene radicals of 2 to about 6 carbons, and R' is a lower hydroxyalkyl radical in an amount sufficient to improve the soil removal power thereof.

18. A detergent composition consisting essentially of a water-soluble higher alkyl aryl sulfonate detergent and a minor proportion up to about 10% by weight of a compound having the formula:

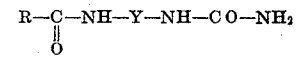


wherein



is a higher fatty acyl radical, and R' is a lower hydroxyalkyl group of 2–3 carbons.

19. A detergent composition consisting essentially of a detergent selected from the class consisting of the water-soluble anionic sulfate and sulfonate detergents, and a compound having the formula:

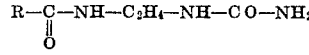


wherein



is a higher fatty acyl radical and Y is selected from the class consisting of alkylene and hydroxyalkylene radicals of 2 to about 6 carbons in an amount sufficient to improve the soil removal power thereof.

20. A detergent composition consisting essentially of a detergent selected from the class consisting of the water-soluble anionic sulfate and sulfonate detergents, and a compound having the formula:



wherein



is lauroyl in an amount sufficient to improve the soil removal power thereof.

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