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Coffindaffer et al.

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[54] **ANTI-DANDRUFF SHAMPOOS WITH PARTICULATE ACTIVE AGENT AND CATIONIC POLYMER**

5,104,645 4/1992 Cardin et al. 514/852
 5,145,607 9/1992 Rich 424/70.28
 5,149,522 9/1992 Schwarz et al. 424/70.28
 5,393,519 2/1995 Dowell et al. 424/70.24

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[57] **ABSTRACT**

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[52] **U.S. Cl.** **424/70.21; 424/70.22; 424/70.24; 424/70.28; 514/880; 514/881**

[58] **Field of Search** 424/70.21, 70.22, 424/70.24, 70.28; 514/880, 881

[56] **References Cited**

U.S. PATENT DOCUMENTS

4,273,760 6/1981 Koehler et al. 424/70.24
 4,298,494 11/1981 Parslow et al. 252/550
 4,830,774 5/1989 LaPetina et al. 514/852
 4,835,148 5/1989 Barford et al. 514/881
 4,997,641 3/1991 Hartnett et al. 424/70.28

Provided is a anti-dandruff shampoo composition comprising: (a) from about 8% to about 40%, by weight, of anionic deterative surfactant; (b) from about 0.1% to about 5%, by weight, of particulate anti-dandruff agent having an average particle size of from about 0.35 microns to about 5 microns; (c) from about 0.01% to about 1.0%, by weight, of a stabilizing agent for said anti-dandruff agent, said stabilizing agent being a soluble cationic polymer; (d) from about 50% to about 91.89%, by weight, water; wherein said shampoo composition is substantially free of suspending agents selected from the group consisting of crystalline suspending agents and anionic, nonionic, and amphoteric polymeric suspending agents. Also provided is a process used for making the present compositions wherein said particulate anti-dandruff agent and said cationic polymer are admixed in water in the presence of anionic deterative surfactant.

17 Claims, No Drawings

**ANTI-DANDRUFF SHAMPOOS WITH
PARTICULATE ACTIVE AGENT AND
CATIONIC POLYMER**

TECHNICAL FIELD

The present invention relates to shampoo compositions containing particulate anti-dandruff agents. In particular, the present invention relates to shampoo compositions containing a particulate anti-dandruff agent and a cationic polymer.

BACKGROUND OF THE INVENTION

Shampoo compositions for cleaning the hair which also contain anti-dandruff agents are well known. Among the preferred types of anti-dandruff agents are particulate, crystalline anti-dandruff agents, such as sulfur, selenium disulfide, and heavy metal salts of pyridinethione. In order for these types of shampoos to be effective and to provide a consistent level of performance, without requiring vigorous shaking of the package in which they are contained, it is conventional practice to suspend them in the composition with the aid of a suspending agent. Since shampoos are likely to remain on shelves or in storage for long periods of time, it is important for the suspending agents to keep the particulate anti-dandruff agents well suspended for relatively long periods of time. The suspending agents which have become preferred for suspension of particulate anti-dandruff agents are those which form a crystalline network in the shampoo when the shampoo is stationary, but which allow the composition to readily flow when shear is applied, such as when a user pours it out of a bottle. Examples of such suspending agents include ethylene glycol distearate and N, N- di- (hydrogenated tallow) amido benzoic acid. Crystalline suspending agents are currently the preferred method for suspending particulate anti-dandruff agents in the marketplace.

Other suspending agents which are known include hydrophilic polymeric thickening agents such as cellulosic gums and acrylic acid/acrylate polymers, the latter of which are commonly referred to as carbomers. Although these materials are effective for suspending particulate matter, at higher levels they tend to impart an undesirable, slimy feel.

Just as important as suspending the anti-dandruff agent, the suspending agent must also allow the anti-dandruff agent to deposit on the scalp during use. If too little anti-dandruff agent deposits, it will be unable to provide good anti-dandruff efficacy. The polymeric suspending agents are believed to be less efficient than the crystalline suspending agents for deposition of active ingredients on the scalp and hair.

Another drawback of crystalline suspending agent is that they require costly heating and cooling steps in the manufacture of the compositions in order to make high quality stable suspensions.

Another important parameter in the formulation of anti-dandruff shampoos is lathering. The consuming public often associates high lathering with effective cleaning, and typically prefers high lathering shampoos to low lathering shampoos from an aesthetic standpoint. Unfortunately, crystalline suspending agents tend to adversely affect lathering performance.

It is also known to prepare antimicrobial compositions utilizing finely powdered polyvalent metal salts of 2-mercaptopyridine-N-oxide, e.g., zinc pyridinethione. See, for example, U.S. Pat. No. 4,832,950 (Takaya et al., issued May 23, 1989) and U.S. Pat. No. 4,670,430 (Inamura et al.,

issued Jun. 2, 1987). In these compositions, very small average particle size of less than 0.2 microns, are said to provide improved dispersion stability without the use of polymeric suspending agents or other means to suspend larger particles. Compared to such prior compositions, the compositions of Imamura et al. are said to provide improved adsorbability. Takaya et al. teaches further suspension stability of the small particles of Imamura et al. via the use of a specific dispersent selected from the group consisting of: (A) polyglycol/polyamine polyglycol/polyamine/alkylamine or alkyleneamine condensation polymers; (B) water soluble polymers selected from the group consisting of hydroxyalkylcelluloses and partly quaternized products thereof, and at least one nonionic surfactant; and (C) at least one cationic polymer compound and at least one inorganic salt.

Although the above compositions avoid the use of conventional suspending agents, deposition efficiency of the particulate metal salts remains impaired due to the extremely small size of these particles. Because of their small size, a proportion of them would tend to remain dispersed in solution rather than deposit on the scalp, skin, or hair.

Thus, it would be desirable to provide liquid topical composition with a stable dispersed anti-dandruff agent, which utilized larger particles than those specified in the above patents, but which also did not require the use of conventional suspending agents such as crystalline suspending agents or polymers added for thickening purposes.

It is an object of this invention to provide anti-dandruff shampoos containing particulate anti-dandruff agents that are suspended without the need for crystalline suspending agents, but which still retain efficient deposition of the anti-dandruff agent on the hair or scalp. It is another object of this invention to provide anti-dandruff shampoos containing particulate anti-dandruff agents that are suspended without the need for hydrophilic polymeric thickening agents, but which still retain efficient deposition of the anti-dandruff agent on the hair or scalp.

It is another object of this invention to provide such compositions, as set forth above, which have good, non-slimy feel. It is yet another object of this invention to provide compositions as set forth above which have improved deposition efficiency on the skin, scalp and/or hair, relative to conventional suspending agents for particulate matter in liquid topical compositions. It is still another object of this invention to provide compositions, as set forth above, which can be made without the need for costly heating and cooling steps, as conventionally utilized when crystalline suspending agents are employed.

It is yet another object of this invention to provide a process for making anti-dandruff shampoos meeting the above objects.

These and other benefits as may be apparent or otherwise realized can be obtained according to the present invention, which is described below.

Unless otherwise indicated, all percentages are calculated by weight of the total composition, and all ratios are calculated on a weight basis. Unless otherwise indicated, ingredients are based on the active level and, therefore do not include carriers or by-products that may be included in commercially available materials. The present invention may comprise, consist of, or consist essentially of any of the essential and various optional and/or preferred ingredients and elements described herein.

SUMMARY OF THE INVENTION

It has now been found that anti-dandruff shampoos meeting the above objects can be achieved. In particular, it has been found that relatively small particle size particulate anti-dandruff agents can be suspended in shampoo compositions containing anionic surfactants and relatively low levels of a shampoo soluble cationic polymer, without the need for crystalline suspending agents or polymeric thickening agents for suspending the particles. The present compositions utilize particles having an average particle diameter of from about 0.35 microns to about 5 microns. By way of theory, and without intending to necessarily limit the invention, it is believed that the cationic polymers hereof form a net-like suspension by bridging miscelles of the anionic surfactant. The small particle size particulate anti-dandruff agent then becomes suspended within this network.

More specifically, the present invention provides an anti-dandruff shampoo composition comprising:

- (a) from about 8% to about 40%, by weight, of anionic deterative surfactant;
- (b) from about 0.1% to about 5%, by weight, of particulate anti-dandruff agent having an average particle size of from about 0.35 microns to about 5 microns;
- (c) from about 0.01% to about 1.0%, by weight, of a stabilizing agent for said anti-dandruff agent, said stabilizing agent being a soluble cationic polymer;
- (d) from about 50% to about 91.89%, by weight, water; wherein said shampoo composition is substantially free of suspending agents selected from the group consisting of crystalline suspending agents and anionic, nonionic, and amphoteric polymeric suspending agents.

It has also been found that the process used for making the present compositions can be critical to stability of the final product. In particular, the particulate anti-dandruff agent and cationic polymer should preferably be combined or admixed with one another only in aqueous conditions in the presence of anionic surfactant. Combining the particulate anti-dandruff agent and cationic polymer under aqueous conditions, in the absence of anionic surfactant can undesirably lead to agglomeration of the particulate anti-dandruff agent.

More specifically, the present invention provides a process for making a stable anti-dandruff shampoo composition comprising preparing a mixture of:

- (a) from about 8% to about 40%, by weight, anionic deterative surfactant;
- (b) from about 0.1% to about 5%, by weight, of particulate anti-dandruff agent having a volume average particle size of from about 0.35 microns to about 5 microns;
- (c) from about 0.01% to about 1%, by weight, of stabilizing agent for said anti-dandruff agent, said stabilizing agent being a shampoo soluble cationic polymer;
- (d) from about 50% to about 91.89%, by weight, water; wherein said particulate anti-dandruff agent and said cationic polymer are admixed in water in the presence of anionic deterative surfactant, and said composition is substantially free of crystalline suspending agents and anionic, amphoteric, and nonionic polymeric suspending agents.

The present invention can provide shampoos with excellent anti-dandruff efficacy, cleansing, and lathering. In addition, the present invention can result in significant cost savings in view of the elimination of conventional suspending agents, which are typically used at higher levels than are the cationic polymers of the present invention, or which in

the case of crystalline suspending agents require separate heating and cooling steps to process the suspending material. In addition, the compositions hereof can provide enhanced lathering and excellent deposition of anti-dandruff actives on the skin, scalp and/or hair.

DETAILED DESCRIPTION OF THE INVENTION

Deterative Surfactant Component

The compositions of the present invention contain an anionic deterative surfactant component, which preferably comprises alkyl sulfate, alkyl ethoxylated sulfate, or a mixture thereof. The compositions hereof can also comprise other types of anionic surfactants, and can additionally contain nonionic and amphoteric surfactants.

The anionic deterative surfactant component will generally be present at a level from about 8% to about 40%, by weight of the composition, preferably from about 10% to about 30%, more preferably from about 12% to about 22%.

Sulfate Surfactants

The compositions hereof will preferably comprise alkyl sulfate, alkyl ethoxylated sulfate, or a mixture thereof. These materials have the respective formulae (I) ROSO_3M and (II) $\text{RO}(\text{C}_2\text{H}_4\text{O})_x\text{SO}_3\text{M}$, wherein R is alkyl or alkenyl of from about 8 to about 30 carbon atoms, x is 1 to 10, and M is H or a soluble salt-forming cation such as ammonium, alkanolamine, such triethanolamine, monovalent metals, such as sodium and potassium, and polyvalent metal cations, such as magnesium and calcium. The cation M, of the anionic surfactant should be chosen such that the anionic surfactant component is water soluble. Solubility of anionic surfactants, in general, will depend upon the particular anionic surfactants and cations chosen. As an aid to determining appropriate mixtures of anionic surfactants, the anionic surfactants should be chosen such that the Krafft temperature is about 15° C. or less, preferably about 10° C. or less, more preferably about 0° C. or less. It is also preferred that the anionic surfactant be soluble in the composition hereof.

Preferably, R has from about 10 to about 18 carbon atoms in both the alkyl and alkyl ethoxylated sulfates. The alkyl ethoxylated sulfates are typically made as condensation products of ethylene oxide and monohydric alcohols having from about 8 to about 24 carbon atoms. The alcohols can be derived from fats, e.g., coconut oil, palm kernel oil, or tallow, or can be synthetic. Such alcohols are preferably reacted with about 1 to about 10, more preferably from about 1 to about 4, most preferably from about 2 to about 3.5, molar proportions of ethylene oxide and the resulting mixture of molecular species having, for example, an average of 3 moles of ethylene oxide per mole of alcohol, is sulfated and neutralized.

Specific examples of alkyl ether sulfates which may be used in the present invention are sodium and ammonium salts of coconut alkyl triethylene glycol ether sulfate; tallow alkyl triethylene glycol ether sulfate, and tallow alkyl hexaoxyethylene sulfate. Highly preferred alkyl ether sulfates are those comprising a mixture of individual compounds, said mixture having an average alkyl chain length of from about 12 to about 16 carbon atoms and an average degree of ethoxylation of from about 1 to about 4 moles of ethylene oxide.

The sulfate surfactant is preferably comprised of a combination of ethoxylated and nonethoxylated sulfates. The

weight ratio of alkyl sulfate to alkyl ethoxylated sulfate is preferably from about 4:1 to about 1:10, more preferably from about 2:1 to about 1:8, even more preferably from about 1:1 to about 1:5, most preferably from about 1:2 to about 1:4. Weight ratios as described above are preferred for their ability to provide optimum combinations of lather, cleaning, and particulate anti-dandruff agent performance. Alkyl sulfates can provide excellent cleaning and lather performance. Alkyl ethoxylated sulfates can provide excellent cleaning performance, are mild to the skin, and can enhance deposition of the particulate anti-dandruff agent relative to alkyl sulfates.

Other Anionic Surfactants

A preferred type of anionic surfactant, especially for use in combination with anionic sulfate surfactants, are the N-acyl amino acid surfactants. N-acyl amino acid surfactants, for purposes hereof, include N-acyl hydrocarbyl acids and salts thereof, such as those represented by Formula III, as follows:



wherein: R¹ is a C₈-C₂₄ alkyl or alkenyl radical, preferably C₁₀-C₁₈; R² is —H, C₁-C₄ alkyl, phenyl, or —CH₂COOM, preferably C₁-C₄ alkyl, more preferably C₁-C₂ alkyl; R³ is —CR⁴— or C₁-C₂ alkoxy, wherein each R⁴ independently is —H or C₁-C₆ alkyl or alkylester, and n is from 1 to 4, preferably 1 or 2; and M is —H or a cation as previously defined, preferably an alkali metal such as sodium or potassium.

A wide variety of N-acyl acid surfactants and their synthesis are described in *Anionic Surfactants, Part II, Surfactant Science Series, Vol. VII*, edited by Warner M. Linfield, Marcel Dekker, Inc. (New York and Basel), 1976; pp 581-617.

Especially preferred are compounds of Formula III wherein R² is methyl and R³ is —CH₂—, and n is 1, which are known as the N-acyl sarcosinates, and acids thereof. Specific examples include lauroyl sarcosinate, myristoyl sarcosinate, cocoyl sarcosinate, and oleoyl sarcosinate, preferably in their sodium and potassium salt forms.

For the purposes of the surfactants described herein, it should be understood that the terms "alkyl" or "alkenyl" include mixtures of radicals which may contain one or more intermediate linkages such as ether or polyether linkages or non-functional substituents such as hydroxyl or halogen radicals wherein the radical remains of hydrophobic character.

Another type of anionic deterative surfactants are aliphatic sulfonates such as represented by the water-soluble salts of the organic, sulfuric acid reaction products of the general formula (IV):



wherein R₁ is chosen from the group consisting of a straight or branched chain, saturated aliphatic hydrocarbon radical having from about 8 to about 24, preferably about 12 to about 18, carbon atoms; and M is a cation, as previously described, subject to the same limitations regarding polyvalent metal cations as previously discussed. Important examples are the salts of an organic sulfuric acid reaction product of a hydrocarbon of the methane series, including iso-, neo-, and n-paraffins, having about 8 to about 24 carbon atoms, preferably about 12 to about 18 carbon atoms and a sulfonating agent, e.g., SO₃, H₂SO₄, oleum, obtained

according to known sulfonation methods, including bleaching and hydrolysis. Preferred are alkali metal and ammonium sulfonated C₁₂-C₁₈ paraffins (e.g. normal and secondary paraffins).

Additional examples of anionic deterative surfactants which come within the terms of the present invention are the reaction products of fatty acids esterified with isethionic acid and neutralized with sodium hydroxide where, for example, the fatty acids are derived from coconut oil; sodium or potassium salts of fatty acid amides of methyl tauride in which the fatty acids, for example, are derived from coconut oil. Other synthetic anionic deterative surfactants of this variety are set forth in U.S. Pat. Nos. 2,486,921; 2,486,922; and 2,396,278.

Still other anionic deterative surfactants are in the class designated as succinates. This class includes such surface active agents as disodium N-octadecylsulfosuccinate; tetrasodium N-(1,2-dicarboxyethyl)-N-octadecylsulfosuccinate; diamyl ester of sodium sulfosuccinic acid; dihexyl ester of sodium sulfosuccinic acid; dioctyl esters of sodium sulfosuccinic acid.

Other suitable anionic deterative surfactants include olefin sulfonates having about 12 to about 24 carbon atoms. The term "olefin sulfonates" is used herein to mean compounds which can be produced by the sulfonation of alpha-olefins by means of uncomplexed sulfur trioxide, followed by neutralization of the acid reaction mixture in conditions such that any sulfones which have been formed in the reaction are hydrolyzed to give the corresponding hydroxy-alkanesulfonates. The sulfur trioxide can be liquid or gaseous, and is usually, but not necessarily, diluted by inert diluents, for example, by liquid SO₂, chlorinated hydrocarbons, etc., when used in the liquid form, or by air, nitrogen, gaseous SO₂, etc., when used in the gaseous form.

The alpha-olefins from which the olefin sulfonates are derived are mono-olefins having about 12 to about 24 carbon atoms, preferably about 14 to about 16 carbon atoms. Preferably, they are straight chain olefins.

In addition to the true alkene sulfonates and a proportion of hydroxy-alkanesulfonates, the olefin sulfonates can contain minor amounts of other materials, such as alkene disulfonates depending upon the reaction conditions, proportion of reactants, the nature of the starting olefins and impurities in the olefin stock and side reactions during the sulfonation process.

A specific alpha-olefin sulfonate mixture of the above type is described more fully in the U.S. Pat. No. 3,332,880, Pfiaumer and Kessler, issued Jul. 25, 1967, incorporated herein by reference.

Another class of anionic deterative surfactants are the betaalkyloxy alkane sulfonates. These compounds have the following formula (V):



where R₁ is a straight chain alkyl group having from about 6 to about 20 carbon atoms, R₂ is a lower alkyl group having from about 1 (preferred) to about 3 carbon atoms, and M is a water-soluble cation as hereinbefore described.

Many additional synthetic anionic surfactants are described in *McCutcheon's Emulsifiers and Detergents, 1989 Annual*, published by M. C. Publishing Co., which is incorporated herein by reference. Also U.S. Pat. No. 3,929,678, Laughlin et al., issued Dec. 30, 1975, discloses many other anionic as well as other surfactant types and is incorporated herein by reference.

Preferred anionic detergent surfactants for use in the present shampoo compositions include ammonium lauryl sulfate, ammonium laureth sulfate, triethylamine lauryl sulfate, triethylamine laureth sulfate, triethanolamine lauryl sulfate, triethanolamine laureth sulfate, monoethanolamine lauryl sulfate, monoethanolamine laureth sulfate, diethanolamine lauryl sulfate, diethanolamine laureth sulfate, lauric monoglyceride sodium sulfate, sodium lauryl sulfate, sodium laureth sulfate, potassium lauryl sulfate, potassium laureth sulfate, sodium lauryl sarcosinate, sodium lauroyl sarcosinate, lauryl sarcosine, cocoyl sarcosine, ammonium cocoyl sulfate, ammonium lauroyl sulfate, sodium cocoyl sulfate, sodium lauroyl sulfate, potassium cocoyl sulfate, potassium lauryl sulfate, triethanolamine lauryl sulfate, triethanolamine laureth sulfate, monoethanolamine cocoyl sulfate, monoethanolamine lauryl sulfate, sodium tridecyl benzene sulfonate, and sodium dodecyl benzene sulfonate.

Amphoteric Surfactants

Amphoteric surfactants can optionally be used in the present compositions and processes. Examples of amphoteric surfactants which can be used in the present invention include those which are broadly described as derivatives of aliphatic secondary and tertiary amines in which the aliphatic radical can be straight or branched chain and wherein one of the aliphatic substituents contains from about 8 to about 18 carbon atoms and one contains an anionic water solubilizing group, e.g., carboxy, sulfonate, sulfate, phosphate, or phosphonate.

The amphoteric surfactant hereof include the imidazolinium amphoteric surfactants such as those depicted by Formula VI:



wherein R¹ is C₈-C₂₂ alkyl or alkenyl, preferably C₁₂-C₁₆, R² is hydrogen or CH₂CO₂M, R³ is CH₂CH₂OH or CH₂CH₂OCH₂CH₂COOM, R⁴ is hydrogen, CH₂CH₂OH, or CH₂CH₂OCH₂CH₂COOM, Z is CO₂M or CH₂CO₂M, n is 2 or 3, preferably 2, M is hydrogen or a cation, such as alkali metal, alkaline earth metal, ammonium, or alkanol ammonium.

Suitable materials of this type are marketed under the tradename MIRANOL and are understood to comprise a complex mixture of species, and can exist in protonated and non-protonated species depending upon pH with respect to species that can have a hydrogen at R². The imidazolinium amphoteric surfactant hereof can be derived via an imidazolinium intermediate. However, it will be recognized by those skilled in the art that it needn't necessarily be derived via an imidazolinium.

Preferred amphoteric surfactants of Formula VII are monocarboxylates and dicarboxylates. Examples of these materials include cocoamphocarboxypropionate, cocoamphocarboxypropionic acid, cocoamphocarboxyglycinate (alternately referred to as cocoamphodiacetate), and cocoamphoacetate.

Specific commercial products providing the imidazolinium derivative component of the present compositions include those sold under the trade names MIRANOL C2M CONC. N.P., MIRANOL C2M CONC. O.P., MIRANOL C2M SF, MIRANOL CM SPECIAL, MIRANOL ULTRA (Miranol, Inc.); ALKATERIC 2CIP (Alkaril Chemicals); AMPHOTERGE W-2 (Lonza, Inc.); MONATERIC CDX-

38, MONATERIC CSH-32 (Mona Industries); REWOTERIC AM-2C (Rewo Chemical Group); and SCHEROTERIC MS-2 (Scher Chemicals).

Amphoteric surfactants also include aminoalkanoates of the formula (VII):



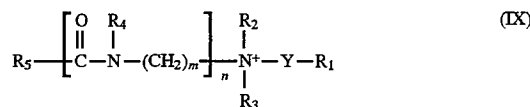
iminodialkanoates of the formula (VIII):



and mixtures thereof; wherein n and m are numbers from 1 to 4, R is C₈-C₂₂ alkyl or alkenyl, and M is hydrogen, alkali metal, alkaline earth metal, ammonium or alkanol ammonium.

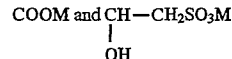
Examples of such amphoteric surfactants include n-alkylaminopropionates and n-alkyliminodipropionates. Such materials are sold under the tradename DERIPHAT by Henkel and MIRATAINE by Miranol, Inc. Specific examples include N-lauryl-beta-amino propionic acid or salts thereof, and N-lauryl-beta-imino-dipropionic acid or salts thereof.

Other amphoteric surfactants that can be used include betaine surfactants such as to be excluded include those represented by the Formula (IX):



wherein:

R₁ is a member selected from the group consisting of



R₂ is C₁-C₃ alkyl or hydroxy (C₁-C₃) alkyl;

R₃ is C₁-C₃ alkyl or hydroxy (C₁-C₃) alkyl;

R₄ is a member selected from the group consisting of hydrogen and C₁-C₃ alkyl;

R₅ is C₈-C₂₀ alkyl or alkenyl;

Y is C₁-C₃ alkyl;

m is an integer from 2 to 7;

n is the integer 1 or 0;

M is hydrogen or a cation, such as an alkali metal or alkaline earth cation metal, ammonium, or alkanolamide.

The term "alkyl" or "hydroxyalkyl" means straight or branch chained, saturated, aliphatic hydrocarbon radicals and substituted hydrocarbon radicals such as, for example, methyl, ethyl, propyl, isopropyl, hydroxypropyl, hydroxyethyl, and the like.

Nonionic Surfactants

Nonionic detergent surfactants can also optionally be used in the present invention. Nonionic surfactants include those broadly defined as compounds produced by the condensation of alkylene oxide groups (hydrophilic in nature) with an organic hydrophobic compound, which may be aliphatic or alkyl aromatic in nature. Examples of preferred classes of nonionic detergent surfactants are:

1. The polyethylene oxide condensates of alkyl phenols, e.g., the condensation products of alkyl phenols having an alkyl group containing from about 6 to about 20

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