



(12) **DEMANDE DE BREVET CANADIEN
CANADIAN PATENT APPLICATION**

(13) **A1**

(86) Date de dépôt PCT/PCT Filing Date: 2011/03/31
(87) Date publication PCT/PCT Publication Date: 2011/10/27
(85) Entrée phase nationale/National Entry: 2012/10/09
(86) N° demande PCT/PCT Application No.: US 2011/030625
(87) N° publication PCT/PCT Publication No.: 2011/133305
(30) Priorité/Priority: 2010/04/19 (US61/325,407)

(51) Cl.Int./Int.Cl. *C11D 11/00* (2006.01),
C11D 1/02 (2006.01), *C11D 1/14* (2006.01),
C11D 1/29 (2006.01), *C11D 1/37* (2006.01),
C11D 11/04 (2006.01), *C11D 17/00* (2006.01),
C11D 3/30 (2006.01)
(71) Demandeur/Applicant:
THE PROCTER & GAMBLE COMPANY, US
(72) Inventeurs/Inventors:
HODSON, STEPHEN JOSEPH, US;
PANCHERI, EUGENE JOSEPH, US
(74) Agent: KIRBY EADES GALE BAKER

(54) Titre : PROCEDE DE FABRICATION D'UNE COMPOSITION DETERGENTE LIQUIDE
(54) Title: PROCESS FOR MAKING A LIQUID DETERGENT COMPOSITION

(57) **Abrégé/Abstract:**

A process for making a laundry liquid detergent comprising anionic deterative surfactant, the process comprising the steps of: a) making a base composition comprising alkanolamine and sodium ions in a molar ratio of from about 0.1 : 1 to 60: 1; and b) adding water to the base composition.



(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
27 October 2011 (27.10.2011)

PCT

(10) International Publication Number
WO 2011/133305 A1

(51) International Patent Classification:

C11D 11/00 (2006.01) *C11D 17/00* (2006.01)
C11D 1/02 (2006.01) *C11D 11/04* (2006.01)
C11D 1/29 (2006.01) *C11D 1/14* (2006.01)
C11D 3/30 (2006.01) *C11D 1/37* (2006.01)

(21) International Application Number:

PCT/US2011/030625

(22) International Filing Date:

31 March 2011 (31.03.2011)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

61/325,407 19 April 2010 (19.04.2010) US

(71) Applicant (*for all designated States except US*): **THE PROCTER & GAMBLE COMPANY** [US/US]; One Procter & Gamble Plaza, Cincinnati, Ohio 45202 (US).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **HODSON, Stephen, Joseph** [US/US]; 3564 Dunlap Court, Mason, Ohio 45040 (US). **PANCHERI, Eugene, Joseph** [US/US]; 7420 Thumbelina Lane, Montgomery, Ohio 45242 (US).

(74) Common Representative: **THE PROCTER & GAMBLE COMPANY**; c/o Eileen L. Hughett, Global Patent

Services, 299 East Sixth Street, Sycamore Building, 4th Floor, Cincinnati, Ohio 45202 (US).

(81) Designated States (*unless otherwise indicated, for every kind of national protection available*): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (*unless otherwise indicated, for every kind of regional protection available*): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report (Art. 21(3))

(54) Title: PROCESS FOR MAKING A LIQUID DETERGENT COMPOSITION

(57) Abstract: A process for making a laundry liquid detergent comprising anionic deterative surfactant, the process comprising the steps of: a) making a base composition comprising alkanolamine and sodium ions in a molar ratio of from about 0.1 : 1 to 60: 1; and b) adding water to the base composition.



WO 2011/133305 A1

PROCESS FOR MAKING A LIQUID DETERGENT COMPOSITION

FIELD OF THE INVENTION

The present invention relates to a process for making a laundry liquid detergent composition. In particular it relates to a process which involves the control of the molar ratio of alkanolamine to sodium ions to achieve a product with a good rheological profile and good stability.

BACKGROUND OF THE INVENTION

Recent liquid laundry detergent consumer preferences towards smaller more concentrated product forms have resulted in the liquid detergent formulators handling a whole series of different constraints. In addition, not only do consumers want smaller compacted liquid laundry detergent products but the consumers also want these compacted products to have the same performance as traditional uncompacted liquid laundry detergents; this is an extremely difficult consumer need to meet.

Compacted liquid laundry detergent products have less space to incorporate detergent ingredients; this places great constraint on the detergent formulator, especially for restricting the levels of the bulk detergent ingredients like surfactants, builders and solvents that take up much of the formulation space. For the detergent ingredients that are incorporated into these compacted liquid laundry detergent products, the detergent formulator must greatly improve the efficiency of these detergent ingredients, and of the compacted liquid laundry detergent composition as a whole.

As well as ensures such compacted liquid laundry detergents have good cleaning performance, the detergent formulator must also ensure that such products have good product storage stability profile, and desirable rheological properties to ensure that the product can be handled and dosed easily by the consumer.

An additional problem associated to compacted detergents is the manufacture process. The reduction of ingredients, such as water and solvents can give rise to undesired phase formation in the base composition, such as surfactant middle phases that are difficult to process.

The aim of the present invention is to overcome the above described drawbacks.

SUMMARY OF THE INVENTION

According to a first aspect of the invention, there is provided a process for making a laundry liquid detergent comprising an anionic deterative surfactant. The process involves the control of the weight ratio of neutralizing agents for the anionic deterative surfactant. In particular it has
5 been found that the specific molar ratio of the process of the invention, gives rise to a favorable rheology profile and to a very stable liquid detergent. The alkanolamine/sodium weight ratio used to neutralize the anionic deterative surfactant plays a crucial role in the control of the rheology of the detergent. The rheology is such that the detergent is neither too runny nor too thick. The detergent is very easy to pour and at the same time thick enough to provide optimum
10 performance during the wash process. Furthermore, the detergent presents good suspending properties providing the opportunity to suspend ingredients without the need of using additional structurants. Another advantage provided by the process of the invention is that can produce detergents with low level of water thereby contributing to compaction.

15 The key step in the process of the invention involves making a base composition comprising anionic surfactant neutralized with an alkanolamine and sodium ions. The molar ratio of alkanolamine, preferably mono-ethanolamine, to sodium ions is from about 0.4:1 to about 60:1, preferably from about 0.5:1 to about 50:1, more preferably from about 1:1 to about 40:1 and especially from about 5:1 to about 35:1. Any alkanolamine can be used in the process of the
20 invention. The alkanolamine can be primary, secondary or tertiary alkanolamine. Preferred alkanolamines include mono-ethanolamine and tri-ethanolamine. Especially preferred for use herein is mono-ethanolamine.

The anionic deterative surfactant preferably comprises an alkyl alkoxylated sulphate. The
25 alkanolamine/sodium ratio of the process of the invention can be achieved, at least in part, by neutralizing part of the alkyl alkoxylated sulphate with an alkanolamine and part of it with sodium ions. Preferably more than 50% and less than 100%, more preferably more than 60% and less than 95% and especially more than 70% and less than 93% of the alkyl alkoxylated sulphate is neutralized with an alkanolamine (preferably with mono-ethanolamine). This contributes not
30 only to a good rheological and stability profile but also to a water reduction in the detergent.

Preferably the alkyl alkoxylated sulphate is pre-neutralized. By “pre-neutralized surfactant” is herein understood a surfactant that has been neutralized before it takes part in the process for making the detergent, as opposite to be neutralized during the process for making the detergent.

Preferably the sulphate deterative surfactant comprises an alkyl ethoxylated sulphate, more preferably a C₈₋₁₈ alkyl ethoxylated sulphate having an average degree of ethoxylation of from 0.5 to 10, preferably from 0.5 to 7, more preferably from 0.5 to 5 and most preferably from 0.5 to 4.

Preferably the anionic deterative surfactant comprises a sulphonate deterative surfactant. In especially preferred embodiments, the alkyl alkoxylated sulphate and the sulphonate deterative surfactant are in a weight ratio of at least about 1.8:1.

10

It is also preferred that the base composition comprises an organic solvent, preferably a non-amino functional solvent. This further reduces the amount of water in the final detergent. Preferred non-amino functional solvents for use herein include alcohols, glycols and mixtures thereof. Especially preferred non-amino functional solvent is a mixture comprising ethanol and propylene glycol and optionally diethylene glycol.

15

Preferably the alkyl alkoxylated sulphate is pre-neutralized and comprises an organic solvent, this again helps to improve the rheology profile of the detergent.

The detergent preferably comprises from 0% to 5%, more preferably less than 2% and especially less than 1% by weight of the detergent of citric acid and any other materials that have a large sphere of hydration associated to them. The detergent delivers similar amount of actives to those delivered in traditional detergents in a smaller dose size and because the water content is lower some chemistry that is not necessarily compatible with or in higher water content products can be made compatible in lower water products.

20
25

Preferably the detergent of the process of the invention comprises a non-ionic surfactant, more preferably in a weight ratio of anionic to non-ionic surfactant of at least 10:1, more preferably at least 20:1.

30

According to a product aspect of the invention, there is provided a laundry detergent obtainable and preferably obtained according to the process of the invention.

The detergent is quite compacted thereby allowing for the delivery of a very small dose (volume

wise) providing good cleaning results. Typical volumes of compacted detergents are below 30 ml, more preferably below 25 ml. The detergent also has a good rheological profile.

According to the last aspect of the invention, there is provided a method of laundering fabric
 5 comprising the step of contacting the fabric in a laundry washing machine with a wash liquor comprising from about 0.2 to about 1 g/l, preferably from about 0.3 to about 0.8 g/l, of the detergent of the invention.

DETAILED DESCRIPTION OF THE INVENTION

10 The present invention envisages a process for making a laundry liquid detergent composition. It also envisages a detergent composition obtainable, preferably obtained, according to the process of the invention, and a method of laundering a fabric using the detergent composition. The process gives rise to a detergent with a good rheological profile and good stability. The detergent
 15 has good physical appearance and good pouring and dispensing behavior. The rheology of the detergent can be such that allows the suspension of particles without the use of an external structurant.

Process for making the base composition

The key step in the process of the invention is to control the molar ratio alkanolamine (preferably
 20 mono-ethanolamine) to sodium ions. The ratio goes from about 0.4:1 to about 60:1, preferably from about 0.5:1 to about 50:1, more preferably from about 1:1 to about 40:1 and especially from about 5:1 to about 35:1.

Preferred anionic surfactants for use herein include sulfate deterative surfactant in particular
 25 alkoxyated and/or un-alkoxyated alkyl sulfate materials.

Preferred alkoxyated alkyl sulfate materials comprises ethoxyated alkyl sulfate surfactants. Such materials, also known as alkyl ether sulfates or alkyl polyethoxylate sulfates, are those which correspond to the formula:

30



wherein R' is a C₈-C₂₀ alkyl group, n is from about 1 to 20, and M is a salt-forming cation. Preferably, R' is C₁₀-C₁₈ alkyl and n is from about 1 to 15. Most preferably, R' is a C₁₂-C₁₆ and n is from about 1 to 6.

The alkyl ether sulfates will generally be used in the form of mixtures comprising varying R' chain lengths and varying degrees of ethoxylation. Frequently such mixtures will inevitably also contain some unethoxylated alkyl sulfate materials, i.e., surfactants of the above ethoxylated
5 alkyl sulfate formula wherein $n=0$. Unethoxylated alkyl sulfates may also be added separately to the compositions of this invention and used as or in any anionic surfactant component which may be present.

Preferred un-alkoxylated alkyl sulfate materials include mid-branched primary alkyl sulfate
10 surfactants having an average carbon chain length of from about 14 to about 17 ("MBAS surfactants"). They provide good cleaning properties. MBAS surfactants with a carbon chain length of about 16 to 17 (also referred to as "HSAS surfactants") generally provide better cleaning than those of other chain-lengths.

15 Preferably the pre-neutralized sulfate deterative surfactant comprises a C_{8-18} alkyl ethoxylated sulphate having an average degree of ethoxylation of from 0.5 to 10, preferably from 0.5 to 7, more preferably from 0.5 to 5 and most preferably from 0.5 to 6. At least 50%, preferably at least 70% and especially 100% of the surfactant has being neutralized with mono-ethanol amine. In some embodiments, the pre-neutralized sulfate deterative surfactant comprises a HSAS
20 surfactant. In other embodiments the pre-neutralized sulfate deterative surfactant comprises a mixture of an alkyl ethoxylated sulphate with a HSAS surfactant, preferably the alkyl ethoxylated sulphate and the HSAS surfactant are in a weight ratio of at least 2:1, more preferably at least 5:1 and specially at least 10:1. Preferably at least 50%, more preferably at least 70% and especially at least 90% of the sulfate deterative surfactant is neutralized with mono-
25 ethanolamine.

Preferably the pre-neutralized sulfate is in the form of a syrup and preferably comprises a non-aminofunctional solvent. As used herein, "non-aminofunctional solvent" refers to any solvent which contains no amino functional groups. Non-aminofunctional solvent include, for example:
30 C1-C8 alkanols such as methanol, ethanol and/or propanol and/or 1-ethoxypentanol; C2-C6 diols; C3-C8 alkylene glycols; C3-C8 alkylene glycol mono lower alkyl ethers; glycol dialkyl ether; lower molecular weight polyethylene glycols; C3-C9 triols such as glycerol; and mixtures thereof. More specifically non-aminofunctional solvent are liquids at ambient temperature and pressure (i.e. 21° C. and 1 atmosphere), and comprise carbon, hydrogen and oxygen. When

present, non-aminofunctional solvent may comprise from about 0% to about 25%, more specifically from about 1 to about 20%, even more specifically from about 5% to about 15% by weight of the syrup. The addition of the non-aminofunctional solvent would contribute to the favorable rheological profile of the detergent composition.

5

Preferred sulphonic deterative surfactant is a C₁₀₋₁₆ alkyl benzene sulfonic acid, preferably C₁₁₋₁₄ alkyl benzene sulfonic acid. Preferably the alkyl group is linear and such linear alkyl benzene sulfonates are known as "LAS". Alkyl benzene sulfonates, and particularly LAS, are well known in the art. Such surfactants and their preparation are described for example in U.S. Patents

10

2,220,099 and 2,477,383.

Detergent composition

Deterative Surfactant

Compositions suitable for use herein comprises from 5% to 70% by weight, preferably from 10% to 60% by weight, more preferably from 20% to 50% by weight, of a certain kind of deterative surfactant component. Such an essential deterative surfactant component must comprise anionic surfactants (preferably sulphate and sulphonic deterative surfactants as described herein before), nonionic surfactants, or combinations of these two surfactant types. Preferably the detergent comprises from about 10% to about 40%, preferably from about 15% to 30% by weight of the detergent of an alkoxylated sulfate deterative surfactant. Preferably the detergent comprises from 5% to 20%, more preferably from 7 to 15% by weight of the detergent of a sulphonate deterative surfactant. Preferably the detergent comprises from 0.1% to 10%, more preferably from 1 to 5% by weight of the detergent of a non-ionic deterative surfactant. Preferably the detergent comprises from 0 to 10%, more preferably from 1 to 5% by weight of the detergent of a fatty acid.

25

Suitable nonionic surfactants useful herein can comprise any of the conventional nonionic surfactant types typically used in liquid detergent products. These include alkoxylated fatty alcohols, ethylene oxide (EO)-propylene oxide (PO) block polymers, and amine oxide surfactants. Preferred for use in the liquid detergent products herein are those nonionic surfactants which are normally liquid.

30

Preferred nonionic surfactants for use herein include the alcohol alkoxylate nonionic surfactants. Alcohol alkoxylates are materials which correspond to the general formula:



wherein R^1 is a $C_8 - C_{16}$ alkyl group, m is from 2 to 4, and n ranges from about 2 to 12. Preferably R^1 is an alkyl group, which may be primary or secondary, that contains from about 9 to 15 carbon atoms, more preferably from about 10 to 14 carbon atoms. Preferably also the alkoxyated fatty alcohols will be ethoxylated materials that contain from about 2 to 12 ethylene
5 oxide moieties per molecule, more preferably from about 3 to 10 ethylene oxide moieties per molecule.

The alkoxyated fatty alcohol materials useful in the liquid detergent compositions herein will frequently have a hydrophilic-lipophilic balance (HLB) which ranges from about 3 to 17. More
10 preferably, the HLB of this material will range from about 6 to 15, most preferably from about 8 to 15. Alkoxyated fatty alcohol nonionic surfactants have been marketed under the tradenames Neodol and Dobanol by the Shell Chemical Company.

Another type of nonionic surfactant which is liquid and which may be utilized in the
15 compositions of this invention comprises the ethylene oxide (EO) - propylene oxide (PO) block polymers. Materials of this type are well known nonionic surfactants which have been marketed under the tradename Pluronic. These materials are formed by adding blocks of ethylene oxide moieties to the ends of polypropylene glycol chains to adjust the surface active properties of the resulting block polymers. EO-PO block polymer nonionics of this type are described in greater
20 detail in Davidsohn and Milwidsky; Synthetic Detergents, 7th Ed.; Longman Scientific and Technical (1987) at pp. 34-36 and pp. 189-191 and in U.S. Patents 2,674,619 and 2,677,700.

Yet another suitable type of nonionic surfactant useful herein comprises the amine oxide surfactants. Amine oxides are materials which are often referred to in the art as "semi-polar"
25 nonionics. Amine oxides have the formula: $R(EO)_x(PO)_y(BO)_zN(O)(CH_2R')_2 \cdot qH_2O$. In this formula, R is a relatively long-chain hydrocarbyl moiety which can be saturated or unsaturated, linear or branched, and can contain from 8 to 20, preferably from 10 to 16 carbon atoms, and is more preferably $C_{12}-C_{16}$ primary alkyl. R' is a short-chain moiety preferably selected from hydrogen, methyl and $-CH_2OH$. When $x+y+z$ is different from 0, EO is ethyleneoxy, PO is
30 propyleneneoxy and BO is butyleneoxy. Amine oxide surfactants are illustrated by $C_{12}-14$ alkyldimethyl amine oxide. Preferably the detergent composition of the invention comprises from about 0.5% to about 5%, more preferably from 0.8% to 3% by weight of the detergent of an amine oxide surfactant.

In the liquid detergent compositions herein, the essential deterative surfactant component may comprise combinations of anionic and nonionic surfactant materials. When this is the case, the weight ratio of anionic to nonionic is at least 2:1, preferably 5:1 and especially 10:1. The detergent composition comprises from 0% to 5%, more preferably less than 2% and especially less than 1% by weight of the detergent of citric acid. It is also preferred that the detergent composition comprises a low level (i.e. from 0% to 5% and preferably below 2% by weight of the detergent) or is free of fatty acid.

10 Preferably the liquid detergent compositions herein have a pH of from about 7 to about 9, more preferably from 8 to 8.5 as measured in 5% aqueous solution at 20°C.

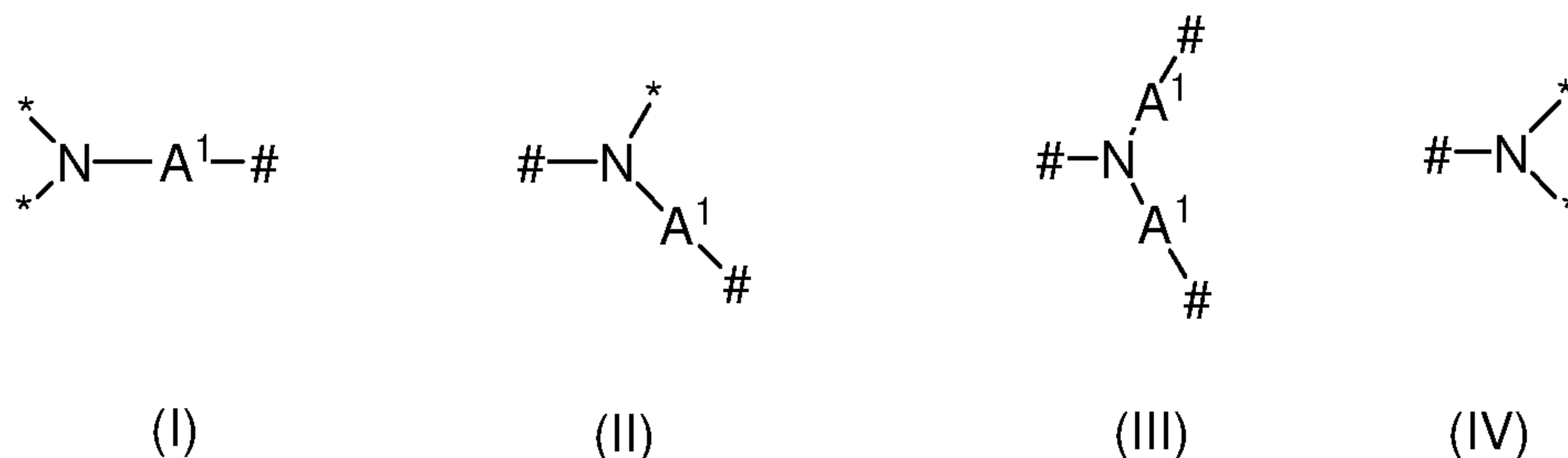
Laundry washing adjuncts

The detergent compositions herein, preferably in liquid form, comprise from 0.1% to 30% by weight, preferably from 0.5% to 20% by weight, more preferably from 1% to 10% by weight, of one or more of certain kinds of laundry washing adjuncts. Such laundry washing adjuncts can be selected from deterative enzymes, builders, chelants, soil release polymers, soil suspending polymers, optical brighteners, dye transfer inhibition agents, bleach, whitening agents, suds suppressors, fabric care benefit agents, solvents, stabilizers, buffers, structurants, dyes and perfumes and combinations of these adjunct types. All of these materials are of the type conventionally utilized in laundry detergent products.

The composition preferably comprises from 1 to 10% by weight of the composition of polymer. Suitable polymers include dispersant polymers such as polyamines, preferably polyethylene imines, most preferably alkoxylated polyethylene imines, preferably the composition comprise from about 1% to about 5% by weight of the composition of an alkoxylated polyethylene imine.

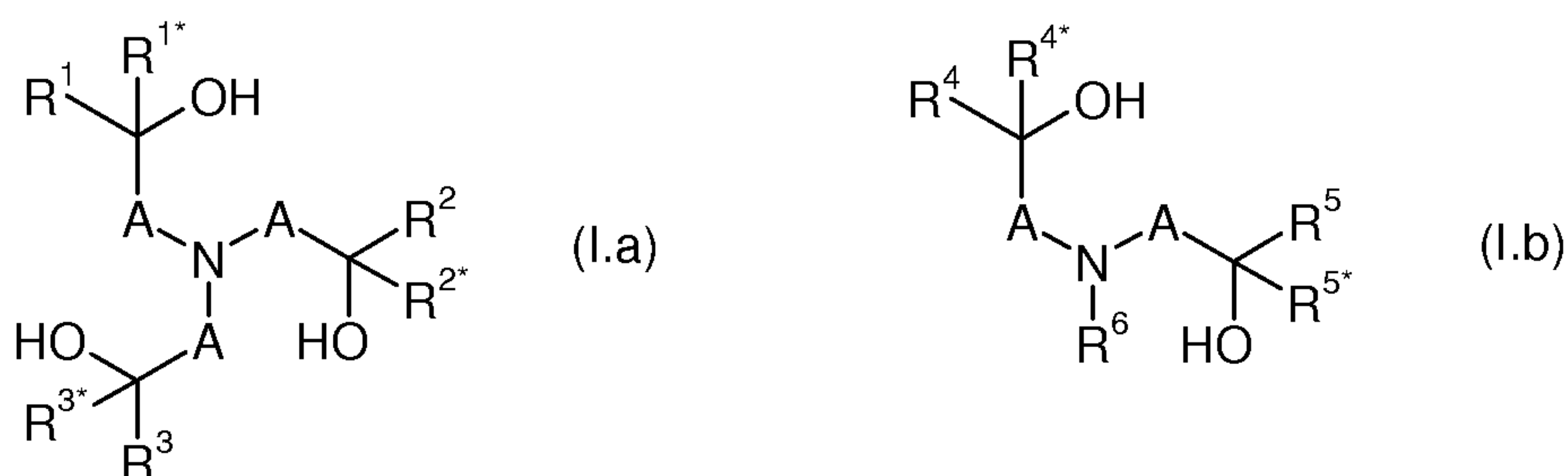
Other preferred polymers include surfactancy boosting polymer. The composition may comprise a surfactancy boosting polymer. Preferred polymers are amphiphilic alkoxylated grease cleaning polymers and/or random graft co-polymers. Amphiphilic alkoxylated grease cleaning polymers refer to any alkoxylated polymers having balanced hydrophilic and hydrophobic properties such that they remove grease particles from fabrics and surfaces. Specific embodiments of the amphiphilic alkoxylated grease cleaning polymers suitable for use herein comprise a core structure and a plurality of alkoxylate groups attached to that core structure.

The core structure may comprise a polyalkylenimine structure comprising, in condensed form, repeating units of formulae (I), (II), (III) and (IV):



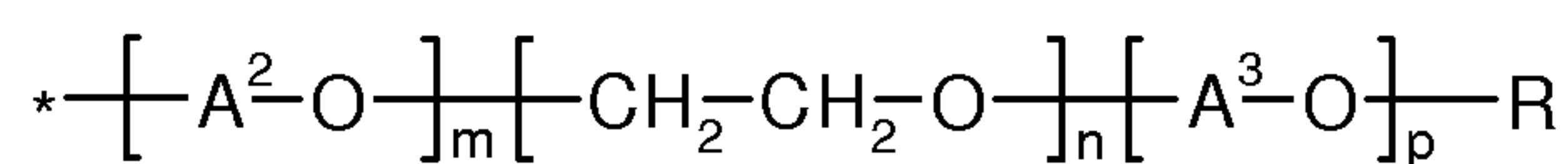
- 5 wherein # in each case denotes one-half of a bond between a nitrogen atom and the free binding position of a group A¹ of two adjacent repeating units of formulae (I), (II), (III) or (IV); * in each case denotes one-half of a bond to one of the alkoxyate groups; and A¹ is independently selected from linear or branched C₂-C₆-alkylene; wherein the polyalkylenimine structure consists of 1 repeating unit of formula (I), x repeating units of formula (II), y repeating units of formula (III)
 10 and y+1 repeating units of formula (IV), wherein x and y in each case have a value in the range of from 0 to about 150; where the average weight average molecular weight, Mw, of the polyalkylenimine core structure is a value in the range of from about 60 to about 10,000 g/mol.

The core structure may alternatively comprise a polyalkanolamine structure of the condensation
 15 products of at least one compound selected from N-(hydroxyalkyl)amines of formulae (I.a) and/or (I.b),



- wherein A are independently selected from C₁-C₆-alkylene; R¹, R^{1*}, R², R^{2*}, R³, R^{3*}, R⁴, R^{4*}, R⁵
 20 and R^{5*} are independently selected from hydrogen, alkyl, cycloalkyl or aryl, wherein the last three mentioned radicals may be optionally substituted; and R⁶ is selected from hydrogen, alkyl, cycloalkyl or aryl, wherein the last three mentioned radicals may be optionally substituted.

The plurality of alkyleneoxy groups attached to the core structure are independently selected from alkyleneoxy units of the formula (V)



(V)

wherein * in each case denotes one-half of a bond to the nitrogen atom of the repeating unit of formula (I), (II) or (IV); A^2 is in each case independently selected from 1,2-propylene, 1,2-butylene and 1,2-isobutylene; A^3 is 1,2-propylene; R is in each case independently selected from hydrogen and C_1 - C_4 -alkyl; m has an average value in the range of from 0 to about 2; n has an average value in the range of from about 20 to about 50; and p has an average value in the range of from about 10 to about 50.

Specific embodiments of the amphiphilic alkoxyated grease cleaning polymers may be selected from alkoxyated polyalkylenimines having an inner polyethylene oxide block and an outer polypropylene oxide block, the degree of ethoxylation and the degree of propoxylation not going above or below specific limiting values. Specific embodiments of the alkoxyated polyalkylenimines suitable for use herein have a minimum ratio of polyethylene blocks to polypropylene blocks (n/p) of about 0.6 and a maximum of about $1.5(x+2y+1)^{1/2}$. Alkoxyated polyalkylenimines having an n/p ratio of from about 0.8 to about $1.2(x+2y+1)^{1/2}$ have been found to have especially beneficial properties.

The alkoxyated polyalkylenimines suitable for use herein have a backbone which consists of primary, secondary and tertiary amine nitrogen atoms which are attached to one another by alkylene radicals A and are randomly arranged. Primary amino moieties which start or terminate the main chain and the side chains of the polyalkylenimine backbone and whose remaining hydrogen atoms are subsequently replaced by alkyleneoxy units are referred to as repeating units of formulae (I) or (IV), respectively. Secondary amino moieties whose remaining hydrogen atom is subsequently replaced by alkyleneoxy units are referred to as repeating units of formula (II). Tertiary amino moieties which branch the main chain and the side chains are referred to as repeating units of formula (III).

Since cyclization can occur in the formation of the polyalkylenimine backbone, it is also possible for cyclic amino moieties to be present to a small extent in the backbone. Such polyalkylenimines containing cyclic amino moieties are of course alkoxyated in the same way as those consisting of the noncyclic primary and secondary amino moieties.

The polyalkylenimine backbone consisting of the nitrogen atoms and the groups A^1 , has an average molecular weight Mw of from about 60 to about 10,000 g/mole, preferably from about 100 to about 8,000 g/mole and more preferably from about 500 to about 6,000 g/mole.

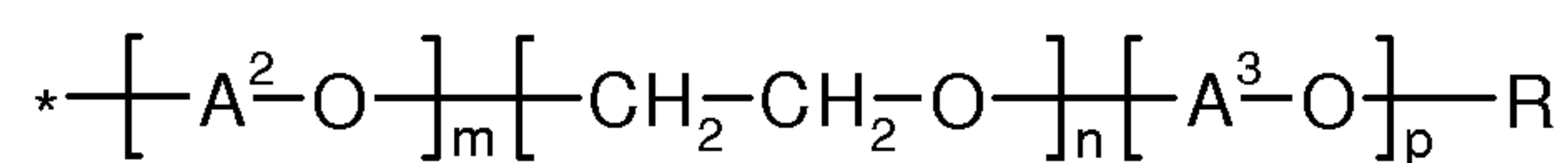
- 5 The sum $(x+2y+1)$ corresponds to the total number of alkylenimine units present in one individual polyalkylenimine backbone and thus is directly related to the molecular weight of the polyalkylenimine backbone. The values given in the specification however relate to the number average of all polyalkylenimines present in the mixture. The sum $(x+2y+2)$ corresponds to the total number amino groups present in one individual polyalkylenimine backbone.

10

The radicals A^1 connecting the amino nitrogen atoms may be identical or different, linear or branched C_2 - C_6 -alkylene radicals, such as 1,2-ethylene, 1,2-propylene, 1,2-butylene, 1,2-isobutylene, 1,2-pentanedyl, 1,2-hexanedyl or hexamethylen. A preferred branched alkylene is 1,2-propylene. Preferred linear alkylene are ethylene and hexamethylene. A more preferred

15 alkylene is 1,2-ethylene.

The hydrogen atoms of the primary and secondary amino groups of the polyalkylenimine backbone are replaced by alkyleneoxy units of the formula (V).



(V)

- 20 In this formula, the variables preferably have one of the meanings given below:

A^2 in each case is selected from 1,2-propylene, 1,2-butylene and 1,2-isobutylene; preferably A^2 is 1,2-propylene. A^3 is 1,2-propylene; R in each case is selected from hydrogen and C_1 - C_4 -alkyl, such as methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl and tert.-butyl; preferably R is hydrogen. The index m in each case has a value of 0 to about 2; preferably m is 0 or approximately 1; more preferably m is 0. The index n has an average value in the range of from about 20 to about 50, preferably in the range of from about 22 to about 40, and more preferably in the range of from about 24 to about 30. The index p has an average value in the range of from about 10 to about 50, preferably in the range of from about 11 to about 40, and more preferably in the range of from about 12 to about 30.

30

Preferably the alkyleneoxy unit of formula (V) is a non-random sequence of alkoxylate blocks. By non-random sequence it is meant that the $[-A^2-O-]_m$ is added first (i.e., closest to the bond to

the nitrogen atom of the repeating unit of formula (I), (II), or (III)), the $[-CH_2-CH_2-O-]_n$ is added second, and the $[-A^3-O-]_p$ is added third. This orientation provides the alkoxyated polyalkylenimine with an inner polyethylene oxide block and an outer polypropylene oxide block.

5

The substantial part of these alkylenoxy units of formula (V) is formed by the ethylenoxy units $-[CH_2-CH_2-O]_n-$ and the propylenoxy units $-[CH_2-CH_2(CH_3)-O]_p-$. The alkylenoxy units may additionally also have a small proportion of propylenoxy or butylenoxy units $-[A^2-O]_m-$, i.e. the polyalkylenimine backbone saturated with hydrogen atoms may be reacted initially with small

10 amounts of up to about 2 mol, especially from about 0.5 to about 1.5 mol, in particular from about 0.8 to about 1.2 mol, of propylene oxide or butylene oxide per mole of NH- moieties present, i.e. incipiently alkoxyated.

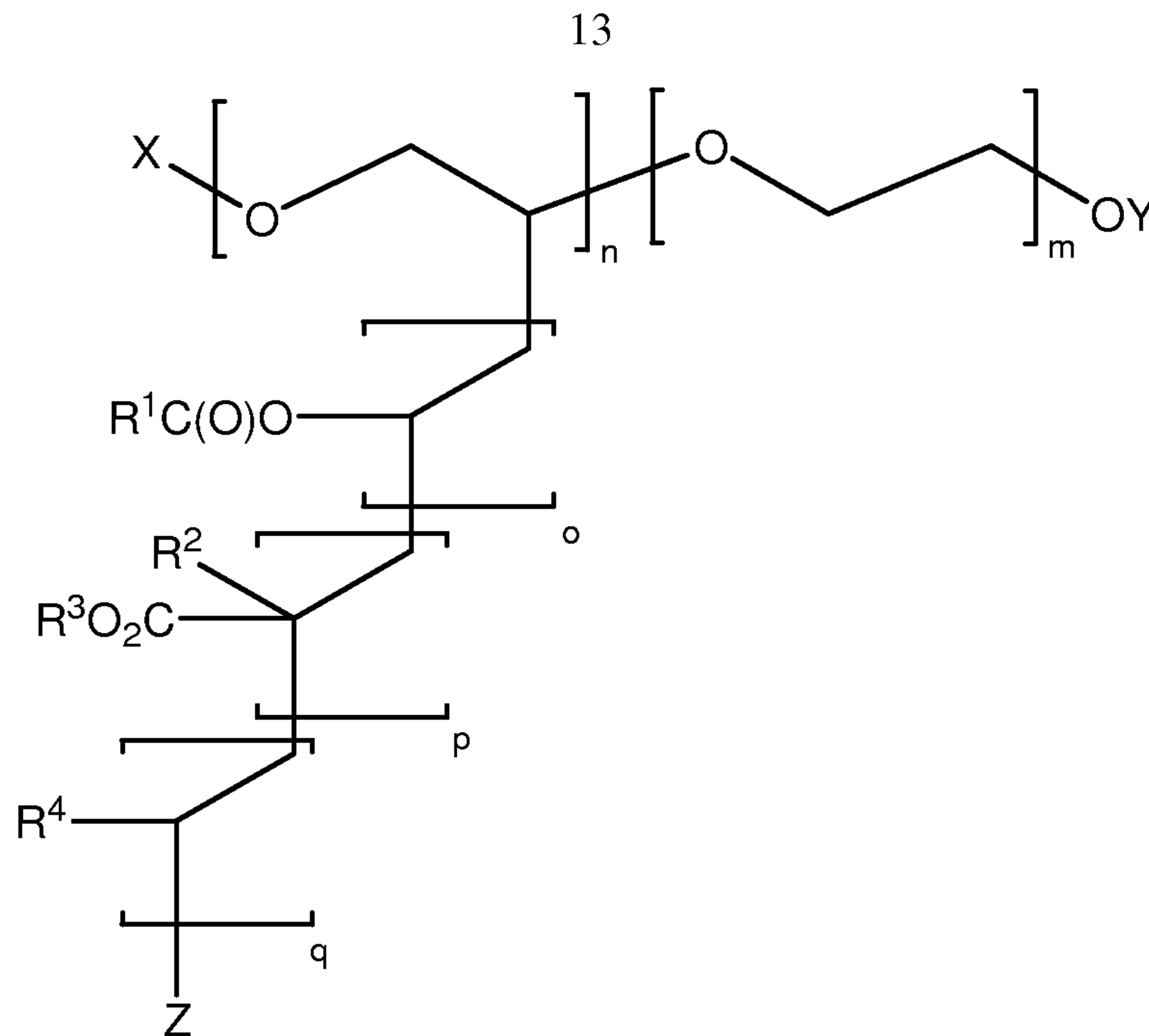
15 This initial modification of the polyalkylenimine backbone allows, if necessary, the viscosity of the reaction mixture in the alkoxylation to be lowered. However, the modification generally does not influence the performance properties of the alkoxyated polyalkylenimine and therefore does not constitute a preferred measure.

20 Preferably the composition comprise from about 0.1% to about 5%, more preferably from about 0.25% to about 2.5% by weight of the composition of an amphiphilic alkoxyated grease cleaning polymer.

25 Suitable random graft co-polymers typically comprise: (i) hydrophilic backbone comprising monomers selected from the group consisting of: unsaturated C_1-C_6 carboxylic acids, ethers, alcohols, aldehydes, ketones, esters, sugar units, alkoxy units, maleic anhydride, saturated polyalcohols such as glycerol, and mixtures thereof; and (ii) hydrophobic side chain(s) selected from the group consisting of: C_4-C_{25} alkyl group, polypropylene, polybutylene, vinyl ester of a saturated C_1-C_6 mono-carboxylic acid, C_1-C_6 alkyl ester of acrylic or methacrylic acid, and mixtures thereof.

30

The polymer preferably has the general formula:



wherein X, Y and Z are capping units independently selected from H or a C₁₋₆ alkyl; each R¹ is independently selected from methyl and ethyl; each R² is independently selected from H and methyl; each R³ is independently a C₁₋₄ alkyl; and each R⁴ is independently selected from pyrrolidone and phenyl groups. The weight average molecular weight of the polyethylene oxide backbone is typically from about 1,000 g/mol to about 18,000 g/mol, or from about 3,000 g/mol to about 13,500 g/mol, or from about 4,000 g/mol to about 9,000 g/mol. The value of m, n, o, p and q is selected such that the pendant groups comprise, by weight of the polymer at least 50%, or from about 50% to about 98%, or from about 55% to about 95%, or from about 60% to about 90%. The polymer useful herein typically has a weight average molecular weight of from about 1,000 to about 100,000 g/mol, or preferably from about 2,500 g/mol to about 45,000 g/mol, or from about 7,500 g/mol to about 33,800 g/mol, or from about 10,000 g/mol to about 22,500 g/mol.

15 Example

Process for making the laundry liquid detergent of the invention:

A 10 liter batch tank with an aspect ratio of about 1.3 (height to diameter) is fitted with an impeller mixer and is charged with the following:

- 20 1.) pre-neutralized sulphate deterative surfactant syrup composed of MEA:C12-15 EO3SO3H, ethanol and propylene glycol
- 2.) pre-neutralized sulphate deterative surfactant syrup composed of MEA:C16-17 Highly Soluble Alkyl Sulfate, ethanol and propylene glycol

14

3.) organic solvent composed of ethanol; propylene glycol and diethylene glycol.

4.) neutralizing agent (mono-ethanolamine)

Stirring is commenced at this point and additions are continued

5

5.) brightener premix composed of brightener chromaphore active, C12-14(EO)₉OH non-ionic surfactant; mono-ethanolamine and water

6.) MEA-Boric acid premix composed of Boric acid, mono-ethanolamine and water

7.) Amine Oxide composed of C12-14 dimethylamine N-oxide and water

10 8.) Ethoxylated Polyamine Dispersant polymer (80 wt% active, 20 wt% water)

9.) Amphiphilic alkoxyated grease cleaning polymer (100% active)

10.) Diethylene triamine penta acetic acid penta sodium salt (DTPA) premix (50 wt% DTPA, 50wt% water)

11.) 1,2-dihydroxybenzene-3,5-disulfonic acid premix (50 wt% active)

15 12.) Calcium formate premix (10 wt%active)

Cooling is applied if needed during the next addition steps to maintain a maximum temperature of less than 37°C

20 13.) C12-18 Fatty acid

14.) C11.8 HLAS (alkyl benzene sulphonate)

Process for making a detergent composition

25 The base detergent is converted to finished detergent by continued stirring and addition of:

15.) water

16.) perfume

17.) Hueing dye premix (0.32% active chromaphore)

30 18.) enzyme premix

19.) Suds supression polymer

The mono-ethanolamine to sodium ion molar ratio of the exemplified composition is about 25:1.

The detergent presents a good rheological profile and it is very stable.

The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as “40 mm” is intended to mean “about 40 mm”.

CLAIMS

What is claimed is:

- 5 1. A process for making a laundry liquid detergent comprising anionic deterative surfactant, the process comprising the steps of:
 - a) making a base composition comprising alkanolamine and sodium ions in a molar ratio of from about 0.1:1 to 60:1; and
 - b) adding water to the base composition.
- 10 2. A process according to claim 1 wherein the anionic deterative surfactant comprises an alkyl alkoxylated sulphate.
- 15 3. A process according to claim 1 or 2 the anionic deterative surfactant comprises a sulphonate deterative surfactant.
- 20 4. A process according to any one of the preceding claim wherein less than 50% of the alkyl alkoxylated sulphate is neutralized with sodium ions.
- 25 5. A process according to any one of the preceding claim wherein at least 50% of the alkyl alkoxylated sulphate is neutralized with an alkanolamine, preferably mono-ethanolamine.
- 30 6. A process according to any of claims 3 to 5 wherein the alkyl alkoxylated sulphate and the sulphonate deterative surfactant are in a weight ratio of at least about 1.8:1.
- 35 7. A process according to any of the preceding claims wherein the base composition comprises an organic solvent.
8. A process according to any one of the preceding claim wherein the detergent comprises from 0% to 5% of citric acid.
9. A process according to any one of the preceding claim wherein the detergent comprises a non-ionic surfactant.
10. A process according to any one of the preceding claim wherein the detergent has a pH of

from about 7 to about 9 as measured in 5% aqueous solution at 20°C.

11. A detergent composition obtainable according to the process of any preceding claim.
- 5 12. A method of laundering fabric comprising the step of contacting the fabric in a laundry washing machine with a wash liquor comprising from about 0.2 to about 1 g/l of a detergent composition according to the preceding claim.