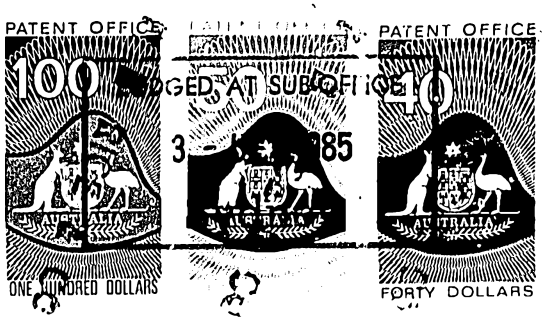
COMMONWEALTH OF AUSTRALIA

596086

Patents Act 1952



CONVENTION APPLICATION FOR A STANDARD PATENT

I/WE, COLGATE-PALMOLIVE COMPANY, a corporation organized and existing under the laws of the State of Delaware, of 300 Park Avenue New York, New York 10022, United States of America.

hereby apply for the grant of a Standard Patent for an invention entitled

CONCENTRATED STABLE NON-AQUEOUS FABRIC SOFTENER COMPOSITION

which is described in the accompanying complete specification.

This application is made under the provision of Part XVI of the Patents Act 1952 and is based on an application for a patent or similar protection made

FEE STAMP TO VALUE OF $ 190 ATTACHED
MAIL OFFICER

in United States of America on 12 December 1984
No. (680,630)

LODGED AT SUB-OFFICE
3 - DEC 1985
Sydney

~~XX~~ ~~OR~~
~~No.~~ (

My/Our address for service is: F.B. RICE & CO.,
28A Montague St,
Balmain N.S.W. 2041

Dated this 29th day of November 1985

COLGATE-PALMOLIVE COMPANY

APPLICATION ACCEPTED AND AMENDMENTS ALLOWED 6.2.90

By: _____
Registered Patent Attorney

TO: The Commissioner of Patents,
COMMONWEALTH OF AUSTRALIA

COMMONWEALTH OF AUSTRALIA

Patents Act

DECLARATION IN SUPPORT OF ~~AN APPLICATION~~ A CONVENTION APPLICATION FOR A PATENT ~~OR PATENT OF ADDITION~~

In support of the ~~Application~~ / Convention Application made by

COLGATE-PALMOLIVE COMPANY
300 Park Avenue
New York, New York 10022
United States of America

for a patent / ~~patent of addition~~ for an invention entitled

CONCENTRATED STABLE NON-AQUEOUS FABRIC SOFTENER COMPOSITION

I/~~We~~ Harold Obstler, Assistant Secretary of an care of the applicant company

do solemnly and sincerely declare as follows:

~~(1) I am/We are the applicant for the patent / patent of addition~~

(1) I am authorised by

the applicant for the patent / ~~patent of addition~~ to make this declaration

on its behalf.

(2) The basic application as defined by Section ~~141~~ / 142 of the Act was / ~~were~~

made in ~~in~~ United States of America on ~~on~~ December 12, 1984

by

Yvon Demangeon 12 Rue Guillaumede de Beaumont Hardicourt, Meulan, France	Michel Julemont Thier de la Fontaine 4 4802 Heusy, Belgium	Marie-Helene Fraikin Rue Saint Lambert, 46 440 Herstal, Belgium

who are the actual inventors of the invention and the facts upon which the applicant company is/~~are~~ entitled to make the application are as follows:

the applicant is the assignee of the invention from the said actual inventors

The basic application referred to in paragraph 2 of this Declaration is/~~are~~ the first application made in a Convention country in respect of the invention the subject of the application.

Declared at NEW YORK NOV 0 8 1985 this 8th day of November 1985

COLGATE-PALMOLIVE COMPANY

By: _____

Harold Obstler, Assistant Secretary

To: The Commissioner of Patents

F. B. RICE & CO.,
Patent Attorneys,
Sydney

(12) PATENT ABRIDGMENT (11) Document No. AU-B-50708/85
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 596086

(54) Title
NON AQUEOUS FABRIC SOFTENER/QUATERNARY AMMONIUM

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(56) Prior Art Documents
AU 548650 90454/82 D06M 13/46
EP 125103
EP 112719

(57) Claim

1. A stable substantially non-aqueous, concentrated fabric softening composition comprising (A) a water dispersible quaternary ammonium compound cationic softener; (B) a non-aqueous carrier liquid comprising hexylene glycol, and (C) up to 15% by weight of a non-ionic surfactant, wherein the composition comprises 20 to 80% by weight of (A) and 80 to 20% by weight of (B).

9. A method of forming a stable, concentrated non-aqueous fabric softening composition according to claim 1, which comprises combining a water dispersible quaternary ammonium compound cationic fabric softening compound with a substantially non-aqueous liquid carrier comprising hexylene glycol, and optionally a nonionic surfactant, at about room temperature, the amount of the cationic compound being at least 20% by weight of the composition.

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Patent Act 1952

COMPLETE SPECIFICATION

(ORIGINAL)

Class Int. Class

Application Number : 50708/85

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Address for Service : F.B. RICE & CO., Patent Attorneys, 28A Montague Street, BALMAIN. 2041. see back hereof for Inventor No. 3/

Complete Specification for the invention entitled:

CONCENTRATED STABLE NON-AQUEOUS FABRIC SOFTENER COMPOSITION

The following statement is a full description of this invention including the best method of performing it known to us:-

CONCENTRATED STABLE NON-AQUEOUS FABRIC SOFTENER COMPOSITION

BACKGROUND OF THE INVENTION

The present invention relates to fabric softener compositions adapted for the use in the rinse cycle of a laundering process and in particular to highly concentrated fabric softener compositions which are easily dispersed in water when used, particularly in laundry machines having automatic dispensing mechanisms.

Compositions containing quaternary ammonium salts having at least one long chain hydrocarbyl group are commonly used to provide fabric softening benefits when employed in a laundry rinse operation; for example, see U.S. Patents 3,349,033; 3,644,203; 3,946,115; 3,997,453; 4,073,735 and 4,119,545.

For most aqueous softener compositions containing cationic quaternary ammonium compounds as active ingredients, the concentration of such cationics has, in general, been limited to the range of about 3 to 6% by weight (see U.S. Patent 3,904,533 and U.S. Patent 3,920,565). Such a low concentration is generally necessitated by the fact that cationics form gels in water systems at concentrations at above about 8%, and while the use of electrolytes to lower the viscosity of such compositions is known (see in particular U.S. Patent 4,199,545), such electrolytes are far from satisfactory. From a functional point of view, the electrolytes often do not perform as required particularly at concentrations of the cationics in the neighborhood of about 12-15%. Further, while the performance of the electrolytes may

mitigate some of the gelling problem, their use is far from satisfactory in providing a highly concentrated aqueous system of cationics which does not gel or severely change in viscosity within the usual range of temperatures encountered in the handling thereof, for example 0°F (about -18°C) up to about 140°F (about 60°C) or in the dispensing from washing machines.

In the ordinary use of European household automatic washing machines, the user places the rinse cycle fabric softener in a dispensing unit (e.g. a dispensing drawer) of the machine. Then, in the operation of the machine, during the rinse cycle, the softener composition is subjected to a stream of cold water to transfer it to the drum. In winter, when the soften composition and the water fed to the dispenser may be especially cold, there can be problems in that some of the composition is not flushed completely off the dispenser during operation of the machine, and a deposit of the composition may build up with repeated wash cycles, so that it may become necessary for the user to flush the dispenser with hot water. This problem can be particularly severe for highly concentrated softener formulations because of the aforementioned gelling problem and also when a nonionic surfactant is present with the cationic softener since there is a tendency for the viscosity of the nonionic to increase when mixed with cold water forming a gel.

In British application 2053249A published February 4, 1981, there are disclosed cationic fabric softening compositions containing 15 to 60% by weight of cationic softener, 25 to 75% by weight of an aqueous medium and 0.5 to 40% by weight of a specified water soluble polymer.

In U.S. Patent 4,351,737 concentrated fabric softeners are described containing both cationic and non-ionic softeners and a non-ionic dispersing agent along with a solvent mixture of a C_1 to C_3 alkanol and a liquid glycol, polyglycol or an alkyl ether thereof. Hexylene glycol is not disclosed.

It has now been found that the dispersibility in cold water and the flow from automatic dispensers, even in highly concentrated, nonionic surfactant-containing liquid fabric softener compositions can be improved considerably by replacing part or all of the coinvention liquid carrier of the non-aqueous liquid softener composition by hexylene glycol.

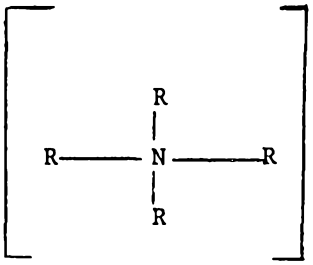
SUMMARY OF THE INVENTION

The present invention provides substantially non-aqueous stable, concentrated softener compositions based upon quaternary ammonium softening compounds and a carrier liquid comprising hexylene glycol and optionally a nonionic surfactant. The present invention also provides a method by which highly concentrated fabric softening compositions are produced and used.

DETAILED DESCRIPTION OF THE INVENTION

The compositions of the present invention are stable substantially non-aqueous compositions which contain a high concentration of the cationic fabric softener which is a water dispersible quaternary ammonium compound as hereinafter described and hexylene glycol as the, or as part of the, liquid carrier. Preferred compositions may also include non-ionic surfactant and electrolyte.

The compositions of this invention contain at least about 20% cationic softener and up to about 80% thereof, preferably up to about 70% and more preferably up to about 60% said cationic softener having the general formula:

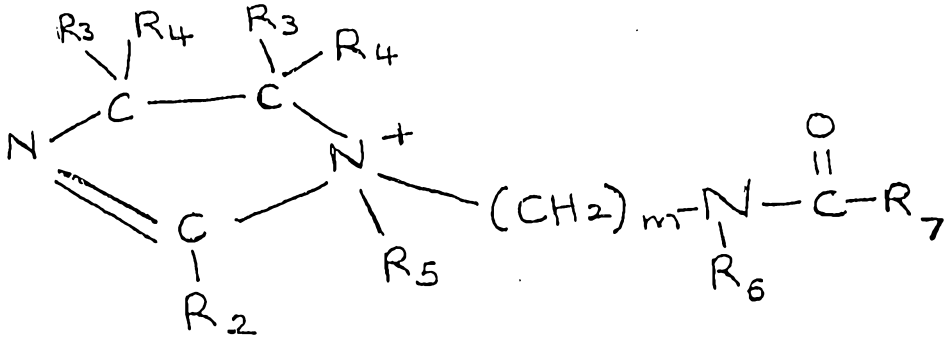
I. 

wherein the R group are selected from C_1 to C_{30} aliphatic, preferably alkyl or alkenyl, aryl (e.g. phenyl, tolyl, cumyl, etc.); aralkyl (e.g. benzyl, phenethyl, etc.); and the halo, amide, hydroxyl, and carboxyl substituents thereof; with the proviso that at least one R is C_{14} to C_{30} and preferably C_{14} to C_{18}, and the others are lower alkyl, and more preferably at least two R's are C_{14} to C_{18} and the others are lower alkyl of C_1 to C_4 (and most preferably methyl or ethyl), or hydroxyalkyl (i.e. $(CH_2\text{-}CH\text{-}O)_xH$ where x is 1 to 10, preferably 1 to 5, most preferably 1 or 2 and R_1 is hydrogen or C_1 to C_4 alkyl, and Y is a water-solubilizing anion such as chloride, bromide, iodide, fluoride, sulfate, methosulfate, nitrite, nitrate, phosphate and carbosylate (i.e. acetate, adipate, propionate, phthalate, benzoate, oleate, etc.). Typical cationics of formula I include the following:

distearyl dimethyl ammonium chloride
ditallow dimethyl ammonium chloride
dihexadecyl dimethyl ammonium chloride
distearyl dimethyl ammonium bromide
di(hydrogenated tallow) dimethyl ammonium bromide
ditallow isopropyl methyl ammonium chloride
distearyl di(isopropyl) ammonium chloride
distearyl dimethyl ammonium methosulfate.

A highly preferred class of cationics is of Formula I wherein two of the R groups are C_{14} to C_{18}, one R is methyl, or ethyl and one R is methyl, ethyl, isopropyl, n-propyl, hydroxyethyl or hydroxypropyl.

Other quaternary softeners which may be used include the imidazolinium compounds of the formula:

II. 

wherein R_2 is C_6 to C_{30}, preferably C_8 to C_{18}, alkyl or alkenyl;

R_3 and R_4 are independently hydrogen or C_1 to C_4 alkyl;

R_5 is C_1 to C_4 alkyl;

R_6 is hydrogen or C_1 to C_4 alkyl;

R_7 is C_6 to C_{30}, preferably C_8 to C_{18} alkyl;

and

m is 2 or 3.

Illustrative compounds of Formula II include:

1-methyl 1-((tallowylamido-) ethyl) -2-talloyl 4,5 dihydro imidazolinium methyl sulfate; 1-methyl-1 ((palmitoylamido) ethyl) 2-octadecyl- 4,5 dihydroimidazolinium chloride.

Specifically preferred cationics are: ditallow monomethyl monohydroxypropyl ammonium chloride, distearyl dimethylammonium chloride, ditallow dimethylammonium chloride, ditallow isopropyl methyl ammonium chloride, etc.

The non-aqueous carrier may comprise from about 80% to about 20%, preferably from about 60% to about 25%, by weight of the composition.

The non-aqueous carrier of the composition of this invention comprises at least about 25% and preferably at least 30% of hexylene glycol and the balance a C_1 to C_3 alkanol, preferably ethanol or propanol, a C_2 to C_6 glycol, preferably diethylene glycol or propylene glycol, or a C_1 to C_6 mono- or di-alkyl ether of such glycols or mixtures thereof. The entire carrier may consist of hexylene glycol to the exclusion of the alcohols and/or glycols (and/or glycol ethers). Hexylene glycol is the commercial name for 2-methylpentane-2,4-diol.

Typical carriers may comprise (a) 25% hexylene glycol and 75% propylene glycol; (b) 31% hexylene glycol and 69% propylene glycol; (c) 31% isopropanol and 69% hexylene glycol; (d) 100% hexylene glycol.

The total amount by weight of the cationic fabric softener and the non-aqueous liquid carrier in the composition will be at least about 80 and up to 100%, preferably from about 85% to 99% by weight, the balance including the nonionic surfactant, electrolyte and/or optional conventional additives.

The optional non-ionic component of the present composition comprises from about 0 to about 15%, preferably from about 1 to 12%, by weight of the composition and generally may vary with the cationic softener in a weight ratio of cationic to nonionic of from about 25:1 to about 3:1, preferably from about 12:1 to 6:1 and especially preferably about 10:1. Suitable

non-ionic compounds include ethylene oxide and propylene (and mixtures thereof) condensates of C_8 to C_{20} aliphatic alcohols and mono, di- and tri-alkyl (each C_4 to C_{12}) phenols. Generally, the non-ionics preferred herein are moderately hydrophilic in nature with a moderate hydrophilic group. In the present invention, this group is preferably an oxyethyl chain generally of at least about 2 terminal oxyethyl groups (i.e. the oxyethyl groups are contigous and terminate in an hydroxyl group) with no more than about 20 such groups, although there may be as many as 200 moles oxyethylene per mole of the hydrophobic group.

Particularly suitable non-ionic surfactants include a C_{9-11} aliphatic alcohol containing 8 moles of condensed ethylene oxide; an ethoxylated nonyl phenol with 6 moles of ethylene oxide, an ethoxylated octyl phenol with 4.5 moles of ethylene oxide and a C_9 aliphatic alcohol with 7.5 moles ethylene oxide.

In addition to the foregoing components of the softening compositions of this invention, there may also be included numerous conventional, supplemental, and optional ingredients which do not adversely affect the stability and/or functional characteristics of the instant compositions. Thus, for example, there may be present the ubiquitous perfumes, dyes, pigments, opacifiers, germicides, optical brighteners, anti-corrosion agents (e.g. sodium silicate), polymers, anti-static agents and the like. Where used, each may comprise, for example, from 0.01% to about 5% by weight of the composition.

It is, of course, recognized and understood that most available chemical materials and particularly those containing an hydrocarbyl moiety are generally mixtures of closely related moieties. Thus, the long chain alkyl substituents (R) in the cationics used in this invention may not only be a single length carbon chain but more likely a mixture. In this regard, a particularly useful quaternary salt, wherein the alkyl groups are derived from tallow, may contain about 35% C_{16} and 60% C_{18} and minor amounts of C_{14} and even others. Similarly, the aliphatic alcohol percursors for the non-ionics used herein may be of a single carbon chain length but more likely, again, it will and can be a mixture in any proportion of the operable chain length compounds.

The fabric softening compositions of this invention should have viscosities in the range of about 10 to 250 centipoises and preferably 25 to 150 centipoises in addition to their essential water-dispersibility in the rinse cycle (or any other form of dilution prior to use).

The manner of combining the hexylene glycol, other optional carriers, cationic softener and optional nonionic surfactant, electrolyte, and other optional conventional additives is not particularly critical, nor is the order of addition. However, some degree of care should be taken to avoid excessive heating in order to limit solvent evaporation and, more importantly, to stay below the flash point temperature of the carrier and any other low boiling liquids that may be present or brought in as components of the softener and the like. For example, the flash point for hexylene glycol is 93°C. Generally, however,

it is preferred to combine all of the ingredients at room temperature, e.g. form about 15°C to 25°C, or lower.

A suitable procedure for preparing the compositions of this invention involves dissolving any nonionic and other additives (e.g. brightener, color, perfume, etc.) in the carrier and then adding this mixture to the cationic which preferably is in liquid form, e.g. as a dispersion in the carrier of any component thereof (for example, as a dispersion in isopropanol).

The following examples will serve to illustrate the present invention without being deemed limitative thereof. Parts are by weight unless otherwise indicated.

EXAMPLE 1

Following the procedure described above, 2 parts of an ethoxylated nonyl phenol (containing 6 moles of ethylene oxide) are dissolved in about 75 parts of hexylene glycol at a temperature of about 20°C. To this solution are slowly added 20 parts of distearyl dimethyl ammonium chloride (75% active in isopropanol) with stirring. A stable product results with a viscosity of about 100 cps.

EXAMPLE 2

The procedure of Example 1 is repeated utilizing the following parts of (A) cationic (active), (B) surfactant, (NaCl); and (C) hexylene glycol

(a) A = 22; B = 2.0; C = 76.0

(b) A = 34; B = 315; C* = 62.5

(c) A = 46; B = 4.8; C = 49.2

(d) A = 58; B = 5.5; C = 36.5

*hexylene glycol/ethylene glycol at 3:1 weight ratio

EXAMPLE 3

Examples 1 and 2 are repeated utilizing in place of distearyl dimethyl ammonium chloride the following:

(a) ditallow dimethyl ammonium chloride

(b) distearyl dimethyl ammonium methosulfate

(c) di(hydrogenated tallow) dimethyl ammonium bromide

(d) di-hexadecyl dimethyl ammonium chloride

(e) distearyl diethyl ammonium chloride.

(f) 1-methyl-1-((tallowylamido)ethyl)-2-tallowyl-4,5-dihydroimidazolinium methyl sulfate.

EXAMPLE 4

In order to demonstrate the improved dispensibility of the hexylene glycol based liquid carrier for the concentrated softening compositions the following formulations A-E are prepared:

	Weight %
Solvent (A-E)	31.9
Cationic fabric softener [1/]	60.0
Nonionic [2/]	5.0
Perfume, dye, minors	3.1

1/ 75% A.I. in isopropanol

2/ nonyl phenol ethoxylated with an average of 6 moles ethylene oxide (C-9 phenol E06:1)

The following are used as the solvent:

A - 10% isopropyl alcohol and 21.9% diethylene glycol (comparison)

B - 10% hexylene glycol and 21.9% propylene glycol

C - 10% isopropyl alcohol and 21.9% hexylene glycol

D - 31.9% hexylene glycol

E - 31.9% propylene glycol (comparison)

Each of the above softener formulations are added to the dispenser of a typical European washing machine. AEG-802, and after 3 cumulative up-to-boil cycles, (the dispenser being refilled for each cycle), the amount of the formulation remaining in the dispenser is measured. The results are shown in the following table as a percent of the total amount used (i.e. a triple dose):

Formulation	Solvent	Quantity Remaining (%)
A	10% isopropanol 21.9% diethylene glycol	15
B	10% hexylene glycol 21.9% propylene glycol	6.7
C	10% isopropanol 21.9% hexylene glycol	3.6
D	31.9% hexylene glycol	2.8
E	31.9% propylene glycol	19

From the above results, it can be easily appreciated that the use of hexylene glycol in replace of all or part of the conventional non-aqueous liquid carriers for rinse cycle added fabric softener formulation greatly improves the cold water dispersibility and flow (dispensibility), even of concentrated formulations containing nonionic surfactants.

Moreover, the concentrated liquid fabric softener compositions of this invention are stable for long periods of time at both low, e.g. 4°C and high, e.g. 35°C storage temperatures, including freeze-thaw cycles, over periods of 6 weeks and longer.

The compositions of this invention have the further advantage that hexylene glycol is non-toxic and, therefore, the formulations are safe to use.

While the instant compositions are intended primarily for direct use in the rinse cycle of automatic washing machines they can also be used in diluted form and for manual treatment, both in and out of the washing machine.

Still further, it should be understood that as used herein, the term "non-aqueous" means that no water is intentionally added to the system, although minor amounts of water, e.g. up to about 5%, can be present, based on addition of specific ingredients, e.g. dyes, as aqueous solutions, the water contained in the softener, etc.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOW:

1. A stable substantially non-aqueous, concentrated fabric softening composition comprising (A) a water dispersible quaternary ammonium compound cationic softener; (B) a non-aqueous carrier liquid comprising hexylene glycol, and (C) up to 15% by weight of a non-ionic surfactant, wherein the composition comprises 20 to 80% by weight of (A) and 80 to 20% by weight of (B).

2. The composition of claim 1, wherein the weight ratio of cationic fabric softener to nonionic surfactant is from 25:1 to 3:1.

3. The composition of claim 1 wherein the non-aqueous carrier (B) comprises at least 25% by weight of hexylene glycol.

4. The composition of claim 1 wherein the non-aqueous carrier (B) comprises at least 35% by weight of hexylene glycol.

5. The composition of claim 1 wherein the non-aqueous carrier consists of hexylene glycol.

6. The composition of claim 1 wherein the non-aqueous carrier comprises a mixture containing at least 25% by weight of hexylene glycol and the balance being a C_1 to C_3 alkanol, a C_2 to C_6 glycol, a C_1 to C_6 mono- or di-alkyl ether of said glycol, or mixtures thereof.

7. The composition of claim 1 wherein said non-aqueous carrier is comprised of (a) 25% hexylene glycol and 75% propylene glycol; (b) 31% hexylene glycol and 69% propylene glycol; (c) 31% isopropanol and 6:% hexylene glycol; or (d) 100% hexylene glycol.

8. The composition of claim 1 wherein the nonionic surfactant is an ethylene of propylene or mixtures thereof oxide condensate of C_8-C_{20} aliphatic alcohol or a mono-, di-, or tri-alkyl phenol, each of said alkyl groups containing from 4 to 12 carbon atoms.

9. A method of forming a stable, concentrated non-aqueous fabric softening composition according to claim 1, which comprises combining a water dispersible quaternary ammonium compound cationic fabric softening compound with a substantially non-aqueous liquid carrier comprising hexylene glycol, and optionally a nonionic surfactant, at about room temperature, the amount of the cationic compound being at least 20% by weight of the composition.

10. A method for imparting softness to fabrics which comprises adding the composition of claim 1 to an aqueous media containing the fabrics in an amount to provide a concentration of the cationic of from 0.005% to 0.5% based on the weight of the fabrics.

11. The method of claim 10 wherein the composition is added during the rinse cycle of an automatic washing machine.

12. The method of claim 11 wherein the composition is added to the aqueous media by flowing a stream of cold water onto the composition whereby the composition is dispersed in the flowing stream and then transferred to the aqueous media.

13. A method for imparting softeners to clothes which combines diluting the composition of claim 1 with from 4 to 15 times as much water by volume as compositions of claim 1 and then adding the diluted composition to the clothes.

DATED this 25 day of January 1989

COLGATE-PALMOLIVE COMPANY
Patent Attorneys for the
Applicant:
F.B. RICE & CO.