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QUATERNARY AMMONIUM SALTS AND FABRIC COATED THEREWITH

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11 Claims. (Cl. 117—139.5)

This application is a continuation-in-part of application Serial No. 608,187, filed September 6, 1956.

The present invention relates to novel quaternary ammonium compounds and methods of preparing the same.

An object of the invention is to provide novel quaternary ammonium salts of fatty acid esters of a polyhydroxypolyamine.

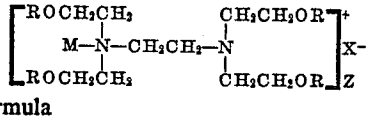
An additional object is to prepare novel compounds useful to impart softening and lubricating characteristics to textiles.

A further object is to prepare novel textile softeners having increased stability to heat.

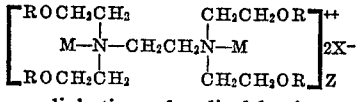
Yet another object is to prepare novel anti-static agents for textiles.

Still further objects and the entire scope of applicability of the present invention will become apparent from the detailed description given hereinafter; it should be understood, however, that the detailed description and specific examples, while indicating preferred embodiments of the invention, are given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this detailed description.

In the parent application it has been disclosed that these objects can be attained by the preparation of quaternary ammonium salts of fatty acid esters of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine having the formula



or the formula



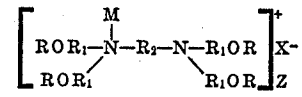
where R is an aliphatic acyl radical having 12 to 18 carbon atoms or hydrogen, at least one R being an aliphatic acyl radical, M is a monovalent hydrocarbon group containing 1 to 4 carbon atoms, X is an anion and Z is the valence of X.

R can be lauroyl, stearoyl, oleoyl, myristoyl, palmitoyl, ricinoleoyl, etc. M can be methyl, ethyl, propyl, isopropyl, n-butyl, isobutyl. X can be a sulfate ion, a halide ion, e. g., chloride, bromide, iodide, benzene sulfonate ion, toluenesulfonate ion, borate ion, phosphate ion, etc. When there are 2 M groups, they can be alike or different. Similarly, when more than one R is an acyl group, the acyl groups can be alike or different.

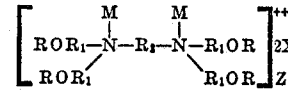
It has now been discovered that in place of the quaternary ammonium salts of the parent application, there can be employed quaternary ammonium salts of fatty acid

esters of polyhydroxyalkyl alkylene polyamines having the formulae:

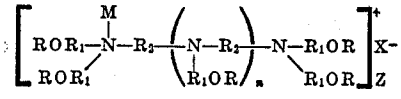
(a)



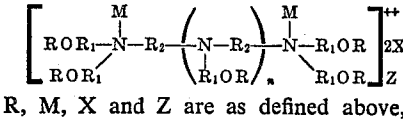
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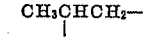
(c)



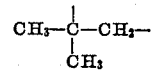
(d)



where R, M, X and Z are as defined above, R_1 is an alkylene group having 2 to 4 carbon atoms, R_2 is an alkylene group having 2 to 6 carbon atoms, and n is an integer from 1 to 3 with the further proviso that in Formula (a) and Formula (b) when R_2 is ethylene, at least one of R_1 has 3 to 4 carbon atoms. The R_1 groups can be the same or different. Similarly, when more than one R_2 groups are present, they can be alike or different. The R_1 and R_2 groups can be straight or branched chain. Thus, R_1 can be one of the groups —CH_2CH_2—,

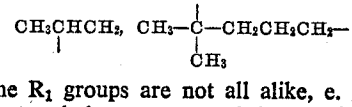


—$CH_2CH_2CH_2$—, —$CH_2CH_2CH_2CH_2$— and

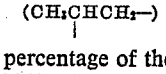


while R_2 can be

—CH_2CH_2—, —$CH_2CH_2CH_2CH_2CH_2CH_2$—



When the R_1 groups are not all alike, e. g., some of the groups are ethylene groups and the remainder are isopropylene



groups, the average percentage of the two types of groups can be from 10 to 90% of ethylene groups and from 90 to 10% of isopropylene groups.

The compounds of the present invention are valuable as softening and anti-static agents for textile fabrics such as cellulose acetate, nylon, vinyl resins, e. g., vinyl chloride resins, etc.

The compounds of the present invention lend valuable softening and lubricity to textile fabric and also prevent the undesirable physical manifestation of static electrical charges which tend to accumulate on many fibers. The compounds are water dispersible which results in easy and low cost application. The compounds also are unique in that they afford a means of binding three or four long fatty acid chains, which when quaternized possess unexpected solubility and compatibility in conventional aqueous textile resin treating baths with internal pH buffering. Such buffering is necessary for urea and melamine formaldehyde resin bath stability.

The present products also have inherent stability to heat (non-scorching) which is superior to the non-quater-

nized acetate salts of the same starting compound, for example.

The compounds of the present invention are made by reacting the appropriate tertiary amine with a quaternizing agent. As quaternizing agents, there can be employed alkyl halides, e. g., methyl bromide, methyl chloride, methyl iodide, ethyl bromide, propyl chloride, n-butyl chloride, dimethyl sulfate, diethyl sulfate, di-n-propyl sulfate, di-n-butyl sulfate, methyl benzene sulfonate, methyl p-toluenesulfonate, ethyl p-toluenesulfonate, butyl p-toluenesulfonate, trimethyl phosphate, tributyl phosphate, dimethyl butyl phosphate, trimethyl borate, allyl chloride, allyl bromide, crotyl chloride, etc.

Whether the mono or diquaternary ammonium salt is formed depends on the proportions of the tertiary amine and the quaternizing agent. Two equivalents of quaternizing agent are required to form the diquaternary compound and only one equivalent to form the monoquaternary compound.

As illustrative compounds of the present invention, there can be mentioned the monoquaternary ammonium salt prepared from N,N,N',N'-tetrakis(2-hydroxyethyl) hexamethylenediamine monostearate and dimethyl sulfate, the corresponding salt from diethyl sulfate, the corresponding salt from methyl bromide, the corresponding salt from di-n-butyl sulfate, the corresponding salt from di-n-propyl sulfate, the corresponding salt from methyl chloride, the corresponding salt from methyl bromide, the corresponding salt from methyl iodide, the corresponding salt from ethyl bromide, the corresponding salt from n-propyl bromide, the corresponding salt from n-butyl bromide, the corresponding salt from isbutyl chloride, the corresponding salt from tributyl phosphate, the corresponding salt from trimethyl phosphate, the corresponding salt from trimethyl borate, the corresponding salt from methyl benzenesulfonate, the corresponding salt from methyl p-toluenesulfonate. In place of the monoquaternary ammonium salt, there can be prepared the corresponding diquaternary compound, e. g., the diquaternary ammonium salt prepared from N,N,N',N'-tetrakis(2 - hydroxyethyl)-hexamethylenediamine monostearate and dimethyl sulfate. Similarly, there can be made the monoquaternary ammonium salt prepared from N,N,N',N'-tetrakis(2 - hydroxyethyl) hexamethylenediamine distearate and dimethyl sulfate, as well as of the other quaternizing agents set forth supra. The diquaternary salt of this compounds, e. g., with dimethyl sulfate, also can be made. Likewise, there can be made the monoquaternary ammonium salt prepared from N,N,N',N'-tetrakis(2 - hydroxyethyl) hexamethylenediamine tristearate and dimethyl sulfate, as well as of the other quaternizing agents set forth supra. The diquaternary salt of this compound, e. g., with dimethyl sulfate, also can be made. Additionally, there can be made the monoquaternary ammonium salt prepared from N,N,N',N'-tetrakis(2 - hydroxyethyl) hexamethylenediamine tetrastearate and dimethyl sulfate, as well as of the other quaternizing agents set forth supra. The diquaternary salt of this compound, e. g., with dimethyl sulfate, also can be made.

Typical examples of other compounds within the present invention include the monoquaternary ammonium salt prepared from N,N,N',N'-tetrakis(2 - hydroxyethyl) trimethylenediamine trilaurate and dimethyl sulfate, as well as the corresponding diquaternary ammonium salt, the monoquaternary ammonium salt prepared from N,N,N',N'-tetrakis(2 - hydroxypropyl) ethylenediamine tetraoleate and methyl bromide, the diquaternary ammonium salt from N,N'-di(2 - hydroxyethyl), N,N'-di(2 - hydroxypropyl) ethylenediamine triricinoleate and methyl p-toluenesulfonate, the monoquaternary ammonium salt from N,N,N',N'-tetrakis(4 - hydroxybutyl) ethylenediamine tristearate and dimethyl sulfate, the monoquaternary ammonium salt prepared from N,N,N',N'',N''-penta (4 - hydroxybutyl) diethylenetriamine distearate and dimethyl sulfate, the diquaternary ammonium salt from N,N,N',N'',N''',N'''',N''''-hepta(2 - hydroxypropyl) tetraethylenepentamine heptastearate and dimethyl sulfate, the monoquaternary ammonium salt from N,N,N',N'-tetrakis(2 - hydroxyethyl) propanediamine - 1,2 trilaurate and allyl chloride, the monoquaternary ammonium salt from N,N,N',N'-tetrakis(2 - hydroxybutyl) propanediamine - 1,3 tristearate and dimethyl sulfate, the monoquaternary ammonium salt from N,N,N',N''-tetrakis(2-hydroxypropyl) hexamethylenediamine tetrastearate and dimethyl sulfate, etc.

It will be appreciated by those skilled in the art that mixed acyl group containing compounds can likewise be prepared and that the acyl group itself can be varied as indicated above.

The starting tertiary amines for the present invention are prepared by ester interchange between the appropriate hydroxyalkyl alkylene polyamine, e. g., N,N,N',N'-tetrakis(2 - hydroxyethyl) hexamethylenediamine, and a lower alkyl ester of a higher fatty acid or fat acid. Mixed esters are prepared by using a mixture of esters, e. g., butyl stearate and butyl laurate in the ester interchange. The ester interchange is catalyzed by alkaline earth and alkali metal hydroxide and alkoxides such as sodium hydroxide, sodium methoxide and also aluminum isopropoxide. The transesterification is carried out either with or without the aid of solvents such as toluene or xylene. The tertiary amine fatty acid ester products are oils or waxes. Increasing the number of acyl groups in the molecule gives a harder wax.

The hydroxyalkyl alkylene polyamine itself is normally prepared by reaction of the appropriate alkylene polyamine, e. g., ethylenediamine or hexamethylenediamine or triethylene tetramine with the appropriate alklene oxide or mixtures of alkylene oxides, e. g., ethylene oxide, propylene oxide, trimethylene oxide, butylene oxide, etc.

Illustrative examples showing the preparation of the materials of the present invention, as well as of the starting materials, are given below.

EXAMPLE 1

N,N,N',N'-tetrakis(β - hydroxyethyl) hexamethylenediamine-1,6 tristearate

A mixture of 73 g. of N,N,N',N'-tetrakis(2 - hydroxyethyl) hexamethylenediamine-1,6 and 255 g. of n-butyl stearate were heated to 100° C. under reduced pressure. The mixture was cooled to 50° C. and 1.2 g. sodium methylate added. The temperature was then increased, and butyl alcohol began to distill from the reaction mixture. The temperature was gradually increased to 140° C. under reduced pressure. The charge was held at 140° C. for thirty minutes to remove the last traces of n-butyl alcohol. The product was a soft wax which contained 1.20 atoms of active hydrogen per mol.

EXAMPLE 2

N,N,N',N'',N''-penta(2-hydroxyethyl) diethylenetriamine tristearate

A stirred mixture of 81 g. of N,N,N',N'',N''-penta(2-hydroxyethyl) diethylenetriamine and 255 g. of n-butyl stearate was heated to 100° C. under reduced pressure. The mixture was cooled to 50° C. and 1.2 g. sodium methylate added. Heat was applied and butyl alcohol began to distill from the reaction mixture. The temperature was increased to 140° C. Heating was continued at 140° C. and under reduced pressure until butyl alcohol no longer distilled. The product was a soft wax.

EXAMPLE 3

N,N,N',N'-tetrakis(2-hydroxypropyl) ethylenediamine tristearate

73 g. of N,N,N',N'-tetrakis(2-hydroxypropyl) ethylenediamine and 255 g. of n-butyl stearate were reacted by

the same procedure used in Example 2 to yield N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine tristearate, a very soft wax.

EXAMPLE 4

N-(2-hydroxyethyl)-N,N',N'-tri(2-hydroxypropyl) ethylenediamine tristearate

In a similar manner to Example 2, 69.5 g. of N-(2-hydroxyethyl)-N,N',N'-tri(2-hydroxypropyl) ethylenediamine was treated with 255 g. of n-butyl stearate to give N-(2-hydroxyethyl)-N,N',N'-tri(2-hydroxypropyl) ethylenediamine tristearate, a very soft wax.

EXAMPLE 5

N,N,N',N'-tetrakis(β-hydroxyethyl) propanediamine-1,2 tristearate

In a manner similar to Example 2, 62.5 g. of N,N,N',N'-tetrakis(β-hydroxyethyl) propanediamine-1,2 and 255 g. n-butyl stearate were reacted to give N,N,N',N'-tetrakis(β-hydroxyethyl) propanediamine-1,2 tristearate as a hard wax.

EXAMPLE 6

N,N,N',N'',N''',N'''',N''''-hepta(2-hydroxyethyl) tetraethylenepentamine tetrastearate

In a similar manner to Example 2, 124 g. of N,N,N',N'',N''',N'''',N''''-hepta(2-hydroxyethyl) tetraethylenepentamine was treated with 340 g. of butyl stearate to give N,N,N',N'',N''',N'''',N'''' - hepta(2 - hydroxyethyl) tetraethylenepentamine tetrastearate.

EXAMPLE 7

N,N,N',N'',N''-penta(2-hydroxyethyl) diethylenetriamine pentastearate

In a similar manner to Example 2, 81 g. of N,N,N',N'', N''-penta(2-hydroxyethyl) diethylenetriamine and 425 g. of butyl stearate were reacted to give N,N,N',N'',N''-penta(2-hydroxyethyl) diethylenetriamine pentastearate.

EXAMPLE 8

N,N,N',N'-tetrakis(β-hydroxyethyl) propanediamine-1,3 tristearate

In a similar manner to Example 2, 62.5 g. of N,N,N', N'-tetrakis(β-hydroxyethyl) propanediamine-1,3 and 255 g. of butyl stearate were reacted to give N,N,N',N'-tetrakis(β-hydroxyethyl) propanediamine-1,3 tristearate.

EXAMPLE 9

Tristearate ester of the amino alcohol formed from the reaction of ethylenediamine with 3.8 mols of ethylene oxide and .2 mol of propylene oxide

In a manner similar to Example 2, 59.8 g. of the amino alcohol formed from the reaction of one mol of ethylenediamine with 3.8 mols of ethylene oxide and .2 mol of propylene oxide was treated with 255 g. of butyl stearate to form the tristearate ester.

EXAMPLE 10

Tristearate ester of the amino alcohol formed from the reaction of ethylenediamine with 3.8 mols of propylene oxide and .2 mol of ethylene oxide

In a similar manner to Example 2, 72.2 g. of the amino alcohol formed from the reaction of one mol of ethylenediamine with 3.8 mols of propylene oxide and .2 mol of ethylene oxide was reacted with 255 g. of butyl stearate to yield the tristearate ester.

EXAMPLE 11

Monoquaternary ammonium salt prepared from N,N,N', N'-tetrakis(β-hydroxyethyl) hexamethylenediamine-1,6 tristearate and dimethyl sulfate

N,N,N',N'-tetrakis(β-hydroxyethyl) hexamethylenediamine-1,6 tristearate (200 g.) was heated to 100° C. and 23.1 g. of dimethyl sulfate added with stirring during one hour. The temperature was maintained at 100° C. for an additional hour. The product was a hard wax which dispersed in water.

EXAMPLE 12

Monoquaternary ammonium salt prepared from N,N,N', N'',N''-penta(2-hydroxyethyl) diethylenetriamine tristearate and dimethyl sulfate

N,N,N',N'',N'' - penta(2 - hydroxyethyl) diethylenetriamine tristearate (200 g.) was heated to 100° C. and 22.9 g. of dimethyl sulfate added during one hour with stirring. After the addition was complete, the whole was maintained at 100° C. for an additional hour. The product was a hard water dispersible wax.

EXAMPLE 13

Monoquaternary ammonium salt prepared from N,N,N', N'-tetrakis(2-hydroxypropyl) ethylenediamine tristearate and dimethyl sulfate

Dimethyl sulfate (23.1 g.) was added during one hour with stirring to 200 g. of N,N,N',N'-tetrakis(2-hydroxypropyl) ethylenediamine tristearate at 100° C. The temperature was maintained at 100° C. for one hour after the addition of the dimethyl sulfate was complete. The product was a hard wax which could be dispersed in water.

EXAMPLE 14

Monoquaternary ammonium salt prepared from N-(2-hydroxyethyl)-N,N',N'-tri(2-hydroxypropyl) ethylenediamine tristearate and dimethyl sulfate

200 g. of N-(2-hydroxyethyl)-N,N',N'-tri(2-hydroxypropyl) ethylenediamine tristearate was heated to 100° C. and 23.4 g. of dimethyl sulfate added during one hour with stirring. The whole was held at 100° C. for one hour after the addition of dimethyl sulfate was complete. The product was a hard water dispersible wax.

EXAMPLE 15

Monoquaternary ammonium salt prepared from N,N, N',N' - tetrakis(β - hydroxyethyl) propanediamine-1,2 and dimethyl sulfate

200 g. of N,N,N',N'-tetrakis(β-hydroxyethyl) propanediamine-1,2 tristearate was heated to 100° C. and 24 g. of dimethyl sulfate added during one hour. The temperature was maintained at 100° C. for an additional hour. The product was a hard wax which dispersed in water.

EXAMPLE 16

Monoquaternary ammonium salt prepared from N,N, N',N'',N''',N'''',N''''-hepta(2-hydroxyethyl) tetraethylenepentamine tetrastearate and dimethyl sulfate

N,N,N',N'',N''',N'''',N''''-hepta(2-hydroxyethyl) tetraethylenepentamine tetrastearate (200 g.) was heated to 100° C. and 16.1 g. of dimethyl sulfate added with stirring during one hour. The temperature was maintained at 100° C. for an additional hour. The product was a hard wax which dispersed in water.

EXAMPLE 17

Diquaternary ammonium salt prepared from N,N,N', N'',N''-penta(2-hydroxyethyl) diethylenetriamine pentastearate and dimethyl sulfate

Dimethyl sulfate (30.5 g.) was added slowly with stirring to 200 g. of N,N,N',N'',N''-penta(2-hydroxyethyl) diethylenetriamine pentastearate at 100° C. during one hour with stirring. The temperature was maintained at 100° C. for one hour after the addition was complete. The product was a hard water dispersible wax.

EXAMPLE 18

Monoquaternary ammonium salt prepared from N,N,N',N'-tetrakis(2-hydroxyethyl) propanediamine-1,3 tristearate and dimethyl sulfate

200 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) propanediamine-1,3 tristearate was heated to 100° C. and 24 g. of dimethyl sulfate added during one hour. The whole was maintained at 100° C. for one hour after the addition was complete. The product was a hard water dispersible wax.

EXAMPLE 19

Monoquaternary ammonium salt prepared from dimethyl sulfate and the tristearate ester of the amino alcohol formed from the reaction of ethylenediamine with 3.8 mols of ethylene oxide and .2 mol of propylene oxide

Dimethyl sulfate (24.3 g.) was added during one hour at 100° C. to 200 g. of the tristearate ester of the amino alcohol formed from the reaction of ethylenediamine with 3.8 mols of ethylene oxide and .2 mol of propylene oxide. The temperature was maintained at 100° C. for one hour after the addition was complete. The product was a hard wax that dispersed in water.

EXAMPLE 20

Monoquaternary ammonium salt prepared from dimethyl sulfate and the tristearate ester of the amino alcohol formed from the reaction of ethylenediamine with 3.8 mols of propylene oxide and .2 mol of ethylene oxide

23.2 g. of dimethyl sulfate was added during one hour at 100° C. to 200 g. of the tristearate ester of the amino alcohol formed from the reaction of ethylenediamine with 3.8 mols of propylene oxide and .2 mol of ethylene oxide. After the addition was complete, the temperature was maintained at 100° C. for one hour. The product was a hard wax that dispersed in water.

EXAMPLE 21

A 0.30% dispersion of the monoquaternary ammonium salt prepared from N,N,N',N'-tetrakis(β-hydroxyethyl) hexamethylenediamine-1,6 tristearate and dimethyl sulfate was applied to "Nylon 200" taffeta in a conventional padding machine at 120° F. in such a manner as to result in 50% pickup of the dispersion by the fabric. The fabric was dried at 225° F. The taffeta possessed improved softness to the hand, increased lubricity and superior anti-scorch and anti-static properties compared to untreated taffeta. "Nylon 200" is a synthetic linear superpolyamide of the hexamethyleneadipamide type.

Unless otherwise stated, all parts and percentages are by weight.

The examples of the parent application are set forth infra.

EXAMPLE 22

N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine monostearate

A mixture of 472 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine, 680 g. n-butyl stearate and 2.9 g. sodium hydroxide was stirred and heated to 110° C. at 70 mm. pressure. n-Butyl alcohol began to distill from the reaction mixture. The temperature was raised to 140° C. during an hour. The pressure was then reduced to 17 mm. and the charge stirred at 140°/17 mm. for one hour and fifteen minutes to remove the last traces of n-butyl alcohol. A total of 151 g. of butyl alcohol was collected (theory 148 g.). The product was a soft tan wax which contained 2.95 atoms of active hydrogen per mol.

EXAMPLE 23

N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate

A stirred mixture of 78.6 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine, 204 g. of n-butyl stearate and 0.7 g. of sodium hydroxide was heated to 120° C. at 70 mm. n-Butyl alcohol began to distill from the reaction mixture. The temperature was raised to 140° C. during one hour. The pressure was then reduced to 20 mm. and the charge stirred at 140° C. for one-half hour. A quantitative yield of product containing 2.0 atoms of active hydrogen per mol was obtained.

EXAMPLE 24

N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine tristearate

A stirred mixture of 236 g. N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine, 1020 g. of n-butyl stearate and 3.1 g. of sodium hydroxide was heated at 70 mm. until n-butyl alcohol began to distill at 106° C. The temperature was maintained at 110° C. and the pressure at 70 mm. for one hour while n-butyl alcohol was collected. The temperature was gradually raised to 140° C. and then the pressure was reduced to 20 mm. The reaction mixture was stirred at these conditions for one and one-half hours. The product was obtained as a wax on cooling.

EXAMPLE 25

N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine tetrastearate

236 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine, 1360 g. of n-butyl stearate and 4 g. of sodium hydroxide were combined and treated by the same procedure used in Example 24 to yield N,N,N',N'-tetrakis-(2-hydroxyethyl) ethylenediamine tetrastearate, a soft wax.

EXAMPLE 26

N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine dioleate

In a similar manner to Example 24, 11.8 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine was treated with 33.8 g. of n-butyl oleate and 0.2 g. of sodium hydroxide to yield N,N,N',N' - tetrakis(2 - hydroxyethyl) ethylenediamine dioleate as a dark viscous liquid.

EXAMPLE 27

N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine dilaurate

In a similar manner to Example 24, 236 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine was treated with 512 g. n-butyl laurate and 1.9 g. of sodium hydroxide to yield N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine dilaurate as a low melting wax.

The compounds of the present invention are prepared as follows:

EXAMPLE 28

Monoquaternary ammonium salt prepared from N,N,N',N' - tetrakis(2 - hydroxyethyl) ethylenediamine monostearate and dimethyl sulfate

N,N,N',N' - tetrakis(2 - hydroxyethyl) ethylenediamine monostearate (150.6 g.) was heated to 100° C. and 37.8 g. of dimethyl sulfate added slowly with stirring. The temperature was maintained between 100° C. and 110° C. and held there for two hours. A soft wax, easily dispersible in water, was obtained.

EXAMPLE 29

Monoquaternary ammonium salt prepared from N,N,N',N' - tetrakis(2 - hydroxyethyl) ethylenediamine monostearate and dimethyl sulfate in aqueous solution

A stirred mixture of 72 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine monostearate and 150 ml. of water was heated to 100° C. and 18 g. of dimethyl sulfate was slowly added. The mixture was heated at

100° C. for five hours and then cooled to room temperature to yield a water dispersible product.

EXAMPLE 30

Monoquaternary ammonium salt prepared from N,N,N', N' - tetrakis(2 - hydroxyethyl) ethylenediamine monostearate and methyl toluenesulfonate

N,N,N',N' - tetrakis(2 - hydroxyethyl) ethylenediamine monostearate (150.6 g.) was heated to 110° C. and 55.8 g. of methyl toluenesulfonate was slowly added with stirring. The temperature was increased to and maintained at 130° C. for four hours. A water dispersible product was obtained.

EXAMPLE 31

Monoquaternary ammonium salt prepared from N,N,N', N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate and dimethyl sulfate

N,N,N',N' - tetrakis(2 - hydroxyethyl) ethylenediamine distearate (153.6 g.) was heated to 130° C. and 25.2 g. of dimethyl sulfate was slowly added. After the addition was complete, the whole was maintained at 130° C. for an additional hour and a half. The product was a water dispersible wax.

EXAMPLE 32

Diquaternary ammonium salt prepared from N,N,N', N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate and dimethyl sulfate

Dimethyl sulfate (50.4 g.) was added slowly with stirring to 153.6 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate at 130° C. The heating was continued after the addition was complete for an additional two hours. The product was a hard wax and may be dispersed in water.

EXAMPLE 33

Monoquaternary ammonium salt prepared from N,N,N', N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate and methyl toluenesulfonate

N,N,N',N' - tetrakis(2 - hydroxyethyl) ethylenediamine distearate (153.6 g.) was heated to 130° C., and 37.2 g. of methyl toluenesulfonate was slowly added with stirring. After the addition was complete, the mixture was heated for four hours at 130° C. The product was a water dispersible wax.

EXAMPLE 34

Monoquaternary ammonium salt prepared from N,N,N', N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate and methyl bromide

Methyl bromide (17.8 g.) was added slowly to 143.5 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate. The addition was started at 63° C., and the mixture was heated to 120° C. during the addition and held at that temperature for an additional thirty minutes. A hard wax was formed that was easily dispersible in water.

EXAMPLE 35

Monoquaternary ammonium salt prepared from N,N,N', N'-tetrakis(2-hydroxyethyl) ethylenediamine tristearate and dimethyl sulfate

206.8 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine tristearate was heated to 127° C., and 25.2 g. of dimethyl sulfate slowly added with stirring. After the addition was complete, the temperature was maintained at 125 to 130° C. for three and one-half hours. The product was a hard wax that dispersed in water. The product calculated for $C_{64}H_{126}N_2O_7$: N, 2.41. Found: N, 2.3. Quaternary ammonium salt 98%; free dimethyl sulfate, none detectable.

EXAMPLE 36

Monoquaternary ammonium salt prepared from N,N,N', N'-tetrakis(2-hydroxyethyl) ethylenediamine dilaurate and dimethyl sulfate

120 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine dilaurate was heated to 65° C. and 25.2 g. of dimethyl sulfate was slowly added with stirring. After all of the dimethyl sulfate had been added, the mixture was heated to 130° C. for three hours. The product was a low melting wax that formed a dispersion with water.

EXAMPLE 37

Monoquaternary ammonium salt prepared from N,N,N', N'-tetrakis(2 - hydroxyethyl) ethylenediamine dioleate and dimethyl sulfate

152.8 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine dioleate was heated to 65° C., and 25.2 g. of dimethyl sulfate slowly added with stirring. After the addition was complete, the temperature was increased to and maintained at 130° C. for three hours. The product was a viscous liquid that formed a dispersion in water.

EXAMPLE 38

Monoquaternary ammonium salt prepared from N,N,N', N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate and allyl chloride

Allyl chloride (15.2 g.) was slowly added to 153 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate which had been warmed to 60° C. After the addition was complete, the whole was heated at 100° C. for one hour to yield the product as a soft, water dispersible wax.

EXAMPLE 39

Monoquaternary ammonium salt prepared from n-butyl bromide and N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate

n-Butyl bromide (27.4 g.) was added slowly to a stirred melt of 153 g. of N,N,N',N'-tetrakis(2-hydroxyethyl) ethylenediamine distearate at 100° C. When the addition was complete, the reaction mixture was stirred and heated at 120° C. for an additional three hours and thirty minutes. The product was a water dispersible wax.

EXAMPLE 40

Monoquaternary ammonium salt prepared from N,N,N', N' - tetrakis(2 - hydroxyethyl) ethylenediamine tetrastearate and dimethyl sulfate

Dimethyl sulfate (6.3 g.) was slowly added to 65 g. of N,N,N',N' - tetrakis(2 - hydroxyethyl) ethylenediamine tetrastearate at 100° C. After the addition was complete, the whole was stirred and heated at 100° C. for one hour to yield a water dispersible wax.

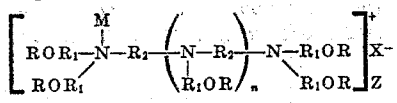
The quaternary ammonium salts of the present invention are water dispersible and possess surface active properties. They are easily applied to fabrics by the usual padding methods from aqueous dispersions. A typical example illustrating this use is as follows:

EXAMPLE 41

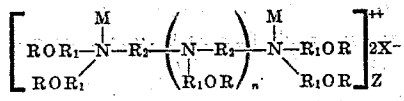
A 0.30% dispersion of the monoquaternary ammonium salt prepared from N,N,N',N' - tetrakis(2-hydroxyethyl) ethylenediamine distearate and dimethyl sulfate was applied to "Nylon 200" taffeta by standard padding techniques, i. .e, a conventional padding machine, at 120° F. in such a manner as to result in 50% pickup of the dispersion by the fabric. The fabric was dried at 225° F. "Nylon 200" taffeta treated in this manner possessed improved softness to the hand, increased lubricity, and superior anti-scorch and antistatic properties. "Nylon 200" is a synthetic linear superpolyamide of the hexamethyleneadipamide type.

We claim:

1. A compound selected from the group consisting of



and



where R is selected from the group consisting of aliphatic acyl radicals containing from 12 to 18 carbon atoms and hydrogen, at least one R being said aliphatic acyl radical; R_1 is an alkylene group having 2 to 4 carbon atoms, R_2 is an alkylene group having 2 to 6 carbon atoms, n is an integer from 0 to 3 with the proviso that when n is 0 and R_2 has 2 carbon atoms at least one R_1 has 3 to 4 carbon atoms, M is a monovalent hydrocarbon group containing 1 to 4 carbon atoms, X is an anion selected from the group consisting of chloride, bromide, iodide, sulfate, benzene sulfonate, toluene sulfonate, phosphate and borate and Z is the valence of X.

2. A compound as recited in claim 1 wherein at least one R is a stearoyl radical.

3. A compound as in claim 1 wherein M is a saturated hydrocarbon group.

4. A compound as in claim 3 wherein X is a sulfate ion.

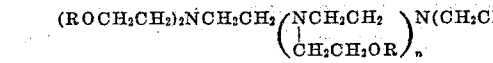
5. A compound as in claim 1 wherein at least one R is a stearoyl radical, M is a saturated hydrocarbon group and X is a sulfate ion.

6. A quaternary ammonium salt of a compound having the formula

$$(ROCH_2CH_2)_2N(CH_2)_nN(CH_2CH_2OR)_2$$

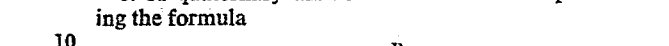
where R is selected from the group consisting of aliphatic acyl radicals containing from 12 to 18 carbon atoms and hydrogen, at least one R being said aliphatic acyl radical, where n is an integer from 3 to 6 and the quaternizing group is a monovalent hydrocarbon group containing 1 to 4 carbon atoms and an anion selected from the group consisting of chloride, bromide, iodide, sulfate, benzene sulfonate, toluene sulfonate, phosphate and borate.

7. A quaternary ammonium salt of a compound having the formula



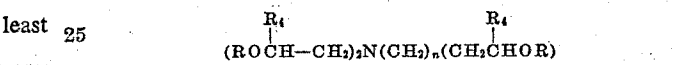
where R is selected from the group consisting of aliphatic acyl radicals containing from 12 to 18 carbon atoms and hydrogen, at least one R being said aliphatic acyl radical, where n is an integer from 1 to 3 and the quaternizing group is a monovalent hydrocarbon group containing 1 to 4 carbon atoms and an anion selected from the group consisting of chloride, bromide, iodide, sulfate, benzene sulfonate, toluene sulfonate, phosphate and borate.

8. A quaternary ammonium salt of a compound having the formula



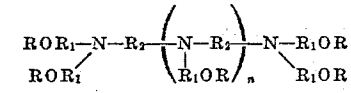
where R is selected from the group consisting of aliphatic acyl radicals containing from 12 to 18 carbon atoms and hydrogen, at least one R being said aliphatic acyl radical, and R_3 is an alkyl group containing 1 to 4 carbon atoms and the quaternizing group is a monovalent hydrocarbon group containing 1 to 4 carbon atoms and an anion selected from the group consisting of chloride, bromide, iodide, sulfate, benzene sulfonate, toluene sulfonate, phosphate and borate.

9. A quaternary amomnium salt of a compound having the formula



where R is selected from the group consisting of aliphatic acyl radicals containing from 12 to 18 carbon atoms and hydrogen, at least one R being said aliphatic acyl radical, R_4 is selected from the group consisting of hydrogen and an alkyl group having 1 to 2 carbon atoms, at least one R_4 being said alkyl group, n is an integer from 2 to 6 and the quaternizing group is a monovalent hydrocarbon group containing 1 to 4 carbon atoms and an anion selected from the group consisting of chloride, bromide, iodide, sulfate, benzene sulfonate, toluene sulfonate, phosphate and borate.

10. A process of preparing a compound set forth in claim 1 comprising reacting a compound having the formula



with a compound having the formula M_2X.

11. A textile fabric having improved softness and antistatic properties coated with the product of claim 1.

References Cited in the file of this patent

UNITED STATES PATENTS

2,719,156 De Benneville et al. ----- Sept. 17, 1955