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(54) Title: PROCESS FOR THE PRODUCTION OF LOW COLOR 2,3-EPOXYPROPYL TRIALKYLAMMONIUM HALIDE			
(57) Abstract An aqueous solution of 2,3-Epoxypropyltrimethylammonium chloride having reduced color and method of preparation are disclosed. 2,3-Epoxypropyltrimethylammonium chloride is treated with sodium hypochlorite to reduce its color. 2-3-Epoxypropyltrimethylammonium chloride can be prepared via reaction of trimethylamine and epichlorohydrin. If desired, an aprotic solvent can be used. 2-3-Epoxypropyltrimethylammonium chloride formed in the resultant slurry, must then be isolated via known methods, such as evaporation, extraction, centrifugation, etc. depending upon the other constituents of the slurry and the residual epichlorohydrin removed. The final product is then treated with an effective amount of sodium hypochlorite. The obtained 2-3-Epoxypropyltrimethylammonium chloride then is used for, for example, color-sensitive applications such as to produce cationic starch for use in paper manufacture.			

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PROCESS FOR THE PRODUCTION OF LOW COLOR
2,3-EPOXYPROPYLTRIALKYLAMMONIUM HALIDE

5

The present invention relates to a process for preparing low color 2,3-epoxypropyltrialkylammonium halide, and more particularly, low color 2,3-epoxypropyltrimethylammonium chloride.

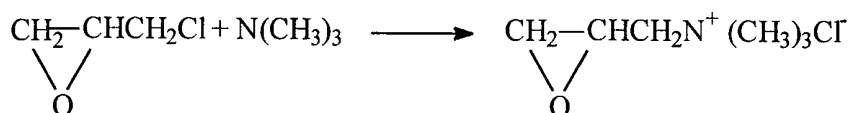
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Various methods for preparing 2,3-epoxypropyltrimethylammonium chloride are known in the art. However, these known processes yield a product which is yellow in color to various degrees. Some of these methods are described hereinafter.

15

James D. McClure, J. Org. Chem., Vol.35, No. 6, pp. 2059-2061 (1970), describes a process for the preparation of glycidyltrimethylammonium chloride by reacting epichlorohydrin with trimethylamine. The reaction is illustrated by the following equation:

20



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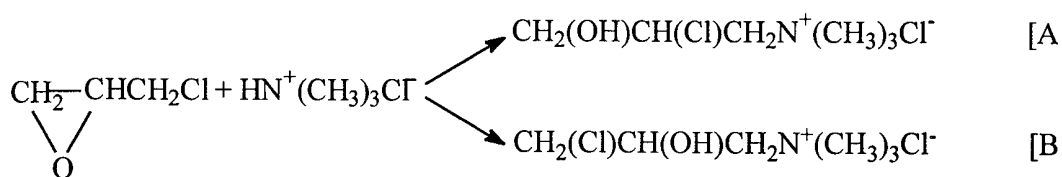
This reaction is best carried out in an aprotic solvent such as ethers, ketones, and esters in which 2,3-epoxypropyltrimethylammonium chloride is insoluble. An excess of epichlorohydrin can be used as solvent as well. Preparation of 2,3-epoxytrimethylpropylammonium bromide by reacting epibromohydrin with trimethylamine is also described in this article.

30

U.S. Patent No. 3,475,458 (J. D. McClure) discloses the preparation of crystalline glycidyltrimethylammonium chloride of high epoxide level, useful in the production of starch ethers, by reacting epichlorohydrin and
 5 trimethylamine in a solvent selected from saturated hydrocarbon ether, lower alkyl ketone, lower alkyl alkanoate and chlorofom-hydrocarbon mixtures.

U.S. Patent No. 4,066,673 (J. B. Doughty et al)
 10 discloses a process for the preparation of 1,2-epoxy propyl trialkylamine chloride in a methanol solution. Epichlorohydrin is first dissolved in methanol and then a stoichiometric amount of trialkylamine (for example, trimethylamine or triethylamine) in methanol is slowly
 15 added thereto.

It is also known to prepare 2,3-epoxypropyltrimethylammonium chloride by a method which involves reacting epoxy compounds, particularly epichlorohydrin, with an
 20 acid salt of a trialkylamine, particularly trimethylamine hydrochloride, in aqueous media, followed by treatment with caustic. This method is illustrated by the following equations:

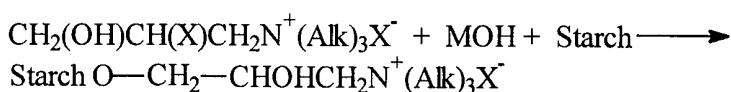
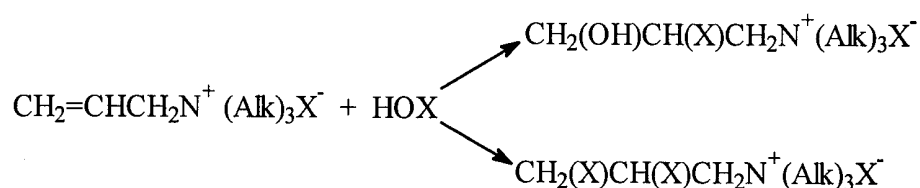
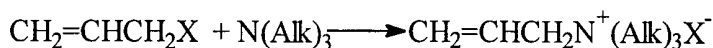


The use of 2,3-epoxypropyltrimethylammonium chloride for preparing cationic starch ethers is also well known. Some of the processes for preparing cationic starch ethers are described hereinafter.

5

U.S. Patent No. 2,876,217 (E. F. Paschall) discloses a process for the preparation of starch ethers containing quaternary ammonium substituents by reacting starch with the reaction product of epihalohydrin and a tertiary amine or a tertiary amine salt. The epihalohydrintriethylamine reaction product is obtained by mixing triethylamine and epihalohydrin with water. After agitating the reaction solution for 5 hours at room temperature, the solution is concentrated to a thick syrup under a vacuum. This epihalohydrintriethylamine syrup is added to an alkaline aqueous slurry of starch. The reaction mass is then neutralized with acid and the cationized starch ether recovered.

U.S. Patent No. 3,346,563 (P. R. Shildneck et al) discloses a process for the preparation of a cationic starch ether by reacting starch and a quaternary halohydrin salt with an alkaline catalyst, the halohydrin being obtained by reacting hypohalous acid with allyl quaternary halide salt of a tertiary amine. This process, is represented by the following equation:



It was postulated that, in the presence of the alkali, an intermediate reaction takes place by which the
 5 halohydroxypropyl quaternary ammonium ether is cycled to form in situ the epoxide and that this epoxide reacts with starch.

2,3-Epoxypropyltrialkylammonium halides are reactive
 10 cationic quaternary amines. They are readily reactive with most substrates, charged or uncharged, to impart to it a permanent cationic charge. Examples of suitable substrate include, for example, natural cellulosic materials such as starch, flour, guar gum, as well as
 15 synthetic materials such as cellulose ethers, polyvinyl alcohol and polymers. In particular, 2,3-epoxypropyltrimethylammonium chloride has been widely commercially used in the manufacture of cationic starches.

20 2,3-Epoxypropyltrialkylammonium halides also suitably impart a permanent cationic charge to various fibers. Suitable fibers include: (i) natural and regenerated cellulosic fibers such as, for example, cotton, linen, and

viscose rayon; (ii) natural and regenerated protein fibers such as, for example, wool, cashmere, and casein; (iii) regenerated fibers such as, for example, cellulose acetate, and cellulose triacetate; (iv) synthetic fibers
5 such as, for example polyamide, polyester, and polyacrylonitrile.

In addition to 2,3-epoxypropyltrialkylammonium halides aqueous solutions of 50% to 70% active 3-chloro-2-
10 hydroxylpropyltrimethylammonium chloride are commercially available for use as quaternizing agents. When this type of the quaternizing agent is used, caustic or other base must be utilized to first convert it to its epoxy form since it is the epoxy form of the quaternizing agent that
15 is the active species for most reactions.

One particular use for these quaternizing agents as reacted with alkaline starch is in the manufacture of paper. The cationic starch is added in the wet-end of a
20 paper process and functions primarily as a dry strength additive. The cationic starch maintains its charge throughout the entire pH range, so that it is usable in acid, neutral, and alkaline paper-making processes. It functions like a charged glue to hold the paper fibers
25 together, and is believed to improve sheet strength by promoting fiber bonding through a combination of ionic bonding with the essentially anionic cellulosic substrates, and also hydrogen bonding. It also finds use in conjunction with internal sizing agents, including
30 alkenyl succinic anhydride ("ASA") and alkylketene dimer ("AKD"), in fine paper applications.

However, in the paper manufacture color and brightness are important qualities. Therefore, raw

materials used in production are desirably colorless or of extremely low color. Unfortunately, widely used conventional direct reaction of epichlorohydrin with trimethylamine produces 2,3-epoxypropyltrimethylammonium chloride which has a strong yellow color. When this product is used to make cationic starch, the yellow color may be imparted to the cationic starch and thus to the final paper product. The color of 2,3-epoxypropyltrimethylammonium chloride can be reduced slightly by lowering the temperature or increasing the ratio of epichlorohydrin to trimethylamine which are reacted to form the 2,3-epoxypropyltrimethylammonium chloride. However, both of these alternatives increase capital and operating costs, and the lower temperatures also slow down the reaction. Increasing the epichlorohydrin to trimethylamine ratio also tends to reduce yield and require more recycle epichlorohydrin for a given reactor.

There is a perceived need in the industry for the 2,3-epoxypropyltrialkylammonium halide product which is devoid of color or has reduced color so that, upon application, less color is imparted to the cationic starch and thence to the paper, or other, final product.

The present invention provides a process for eliminating or reducing the color of 2,3-epoxypropyltrialkylammonium halide prepared by conventional methods.

In one embodiment the present invention is a process for eliminating or reducing color of an aqueous solution of 2,3-epoxypropyltri-alkylammonium halide which process comprises treating an aqueous solution of colored 2,3-epoxypropyltrialkylammonium halide with an effective

amount of an alkali metal salt of hypochlorous acid,
hypochlorous acid or chlorine.

In another embodiment, the present invention is an
5 aqueous solution of 2,3-epoxypropyltrialkylammonium halide
having clear or reduced color prepared by treating an
aqueous solution of colored 2,3-epoxypropyltrialkyl-
ammonium halide with an effective amount of an alkali
metal salt of hypochlorous acid, hypochlorous acid, or
10 chlorine.

In another embodiment, the present invention is a
process for producing a cationic substrate by reacting a
substrate with an aqueous solution of 2,3 epoxypropyl-
15 trialkylammonium halide prepared by the process described
herein-before.

Yet in another embodiment, the present invention is a
cationic substrate prepared by the steps of (1) treating
20 an aqueous solution of colored 2,3-epoxypropyltrialkyl-
ammonium halide with an effective amount of an alkali
metal salt of hypochlorous acid, hypochlorous acid, or
chlorine to reduce its color; and (2) reacting so obtained
aqueous solution of 2,3-epoxypropyltrialkylammonium halide
25 with a substrate to form a cationic substrate.

Yet in another embodiment, the present invention is a
process for producing a cationic fiber by reacting a fiber
with an aqueous solution of 2,3 epoxypropyltrialkyl-
30 ammonium halide prepared by the process described
hereinbefore.

Still in another embodiment, the present invention is
a cationic fiber prepared by the steps of (1) treating an

aqueous solution of colored 2,3-epoxypropyltrialkyl-
ammonium halide with an effective amount of an alkali
metal salt of hypochlorous acid, hypochlorous acid, or
chlorine to reduce its color; and (2) reacting so obtained
5 aqueous solution of 2,3-epoxypropyltrialkylammonium halide
with a fiber to form a cationic fiber.

The present invention provides a simple, convenient
and relatively inexpensive way to obtain 2,3-epoxypropyl-
10 trialkylammonium halide which is essentially clear (water-
white) in color or has reduced color and therefore is
highly desirable for application in the papermaking
industry, as well as in other applications requiring low
color.

15

An aqueous solution of colored 2,3-epoxypropyltri-
alkylammonium halide is treated with an effective amount
of an alkali metal salt of hypochlorous acid, hypochlorous
acid, or chlorine to eliminate or reduce its color.

20

Any alkali metal salt of hypochlorous acid can be
used in the present invention; however, sodium
hypochlorite is preferred. Chlorine can be used either as
a gas or as an aqueous solution.

25

In the context of the present invention, an effective
amount of an alkali metal salt of hypochlorous acid,
hypochlorous acid or chlorine is an amount which is
capable of providing from 200 to 15,000, preferably from
30 400 to 10,000, more preferably from 600 to 5,000, most
preferably from 600 to 2,500, parts per million (ppm) of
active chlorine in the solution.

While it is preferable to add an alkali metal salt of hypochlorous acid, hypochlorous acid, or chlorine to the aqueous solution of 2,3-epoxypropyltrialkylammonium halide at approximately room temperature for the sake of
5 convenience, it is possible to use temperatures from 0 °C to 80 °C and pressures from 0 to 100 psig.

2,3-Epoxypropyltrialkylammonium halide is conveniently prepared by a known process comprising the
10 steps of (1) reacting trialkylamine with epihalohydrin to form a slurry containing solid 2,3-epoxypropyltrialkylammonium halide; (2) extracting 2,3-epoxypropyltrialkylammonium halide from the slurry using water; and (3) removing residual amounts of epihalohydrin from the
15 aqueous solution of 2,3-epoxypropyltrialkylammonium halide. This product has yellow color.

Raw materials (epihalohydrin and trialkylamine) for this process are generally commercially available and
20 reaction conditions are relatively mild, yet yields are high.

The ratio of the trialkylamine to the epihalohydrin is preferably stoichiometric or with an excess of
25 epihalohydrin to serve as a solvent. If a stoichiometric or less than stoichiometric ratio of trialkylamine to epihalohydrin is selected, an additional aprotic liquid is desirably used as a solvent.

30 Any aprotic liquid can be used in the above process. Conveniently, the aprotic liquid used in the above process includes acetone, tetrahydrofuran, dimethoxyethane, ethylacetate, and mixtures thereof.

Epibromohydrin, epichlorohydrin, epiflourohydrin and
epiiodohydrin are all useful in the above process.
Epichlorohydrin is preferred epihalohydrin.

- 5 Any known trialkylamine can be used in the above
process. The trialkylamine can contain same or different
alkyl groups. Preferred are those which contain same
alkyl groups. Trialkylamines in which the alkyl group
contains from 1 to 6 carbon atoms are preferred.
10 Trimethylamine and triethylamine are further preferred
trialkylamines.

The order of addition of the materials is not
important, but if excess epihalohydrin is used to
15 comprehend both reactant and solvent, it is preferable to
charge that component to the reactor first, and then add
the trialkylamine thereafter with stirring. Reaction
temperature is normally from -10 °C to 50 °C. Reaction
time can vary from 1 hour to 48 hours.

20

Once this reaction has taken place a slurry of the
reaction product in the solvent is formed. The reaction
product is 2,3-epoxypropyltrialkylammonium halide.

- 25 This product is then extracted, preferably by
dissolving it in water. Water is preferably added in an
amount to give a 70 percent concentration of 2,3-epoxy-
propyltrialkylammonium halide, with the resultant
formation of two liquid phases. Classic extraction
30 methodology yields a recovery of at least 90 percent,
preferably at least 95 percent, and most preferably 100
percent, of aqueous 2,3-epoxypropyltrialkylammonium
halide, based on theoretical yield. This aqueous phase
has a strong yellow color. The other phase is the

solvent, which may be excess epihalohydrin or other aprotic liquid as defined hereinabove.

5 The resultant aqueous solution of 2,3-epoxypropyltri-alkylammonium halide contains also unreacted epihalohydrin which, if not removed, can be the source of problems in subsequent production of in a finished cationic substrate product. Consequently, this residual epihalohydrin is removed from the solution of 2,3-epoxypropyltrialkyl-
10 ammonium halide by conventional methods such as solvent extraction or distillation.

Thus obtained aqueous solution of colored 2,3-epoxy-propyltrialkylammonium halide is then treated with a small
15 amount of alkali metal salt of hypochlorous acid, hypochlorous acid, or chlorine to eliminate or reduce its color according to the process of the present invention.

2,3-Epoxypropyltrialkylammonium halide aqueous
20 solution produced in the method of the present invention is generally clear (water-white) in color or has reduced color. Color is measured by means of ASTM E 1209 (1993), termed as "Platinum-Cobalt Color, Test Method D 1209". This test method describes a procedure for the visual
25 measurement of the color of essentially light colored liquids and is applicable only to materials in which the color-producing bodies present have light absorption characteristics nearly identical with those of the platinum-cobalt color standards used.

30

2,3-Epoxypropyltrimethylammonium halide, and particularly 2,3-epoxypropyltrimethylammonium chloride, aqueous solution obtained by the process of the present invention is useful for the production of cationic fibers

and cationic substrates for color-sensitive applications such as, for example, the production of cationic starch for use in paper manufacture.

5 Useful substrates which can be imparted permanent cationic charge with 2,3-epoxypropyltrimethylammonium halide aqueous solution prepared according to the present invention include, but are not limited to, natural
10 cellulosic materials such as starch, flour, guar gum, as well as synthetic materials such as cellulose ethers, polyvinyl alcohol and polymers.

 Non-limiting examples of fibers which can be imparted permanent cationic charge with 2,3-epoxypropyltrimethyl-
15 ammonium halide prepared according to the present invention include: (i) natural and regenerated cellulosic fibers such as, for example, cotton, linen, and viscose rayon; (ii) natural and regenerated protein fibers such as, for example, wool, cashmere, and casein; (iii)
20 regenerated fibers such as, for example, cellulose acetate, and cellulose triacetate; (iv) synthetic fibers such as, for example polyamide, polyester, and polyacrylonitrile.

25 The following examples are provided to more fully illustrate the present invention but are not intended to be, nor should they be construed as being, limiting in any way of the scope of the invention.

30 Preparation of 2,3-epoxypropyltrimethylammonium chloride

Step (1): 165 g of epichlorohydrin is charged to a Parr reactor and cooled to 13 °C. Thereafter 21 g of

trimethylamine is added with stirring and the reactants are allowed to react for six hours.

Step (2): To the product obtained in step (1) above
5 is then added 23 g of water, resulting in two liquid
phases. One phase is aqueous solution of 2,3-epoxypropyl-
trimethylammonium chloride, which has strong yellow color,
measured as Platinum-Cobalt Color, Test Method D 1209, of
132. About 65 g of this aqueous phase is recovered. Also
10 recovered is 139 g of excess epichlorohydrin.

Step (3): The aqueous solution of 2,3-epoxypropyl-
trimethylammonium chloride obtained in step (2) above is
extracted with dichloromethane at 1:1 weight ratio six
15 times to remove residual amounts of epichlorohydrin
therefrom. The resulting aqueous solution of 2,3-epoxy-
propyltrimethylammonium chloride is strong yellow in
color.

20

Example 1

Sodium hypochlorite (0.1199 grams, 13 per cent active
chlorine), providing about 575 ppm of active chlorine, is
added, with stirring, to the aqueous solution of 2,3-
25 epoxypropyltrimethylammonium chloride (26.978 grams)
prepared as described hereinbefore and having the yellow
color of 132 measured as Platinum-Cobalt Color, Test
Method D 1209. Almost immediately, the Platinum-Cobalt
Color, Test Method D 1209, is reduced to a reading of 9.
30 Liquid chromatographic scans of the 2,3-epoxypropyl-
trimethylammonium chloride aqueous solution, taken before
and after addition of sodium hypochlorite, show no
degradation of the product.

Example 2

The procedure of Example 1 above is repeated except that 3.326 grams of 0.47 per cent aqueous chlorine solution (providing about 532 ppm of active chlorine) is added to the aqueous solution of 2,3-epoxypropyltrimethylammonium chloride (26.061 grams) prepared as described hereinbefore and having the yellow color of 134 measured as Platinum-Cobalt Color, Test Method D 1209. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyltrimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 22.

Example 3

The procedure of Example 1 above is repeated except that 0.278 grams of 4.2 per cent hypochlorous acid (providing about 582 ppm of active chlorine) is added to the aqueous solution of 2,3-epoxypropyltrimethylammonium chloride (26.817 grams) prepared as described hereinbefore and having the yellow color of 166 measured as Platinum-Cobalt Color, Test Method D 1209. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyltrimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 20.

Example 4

Sodium hypochlorite (0.040 gram, 13 per cent active chlorine), providing about 196 ppm of active chlorine, is added, with stirring, to the aqueous solution of 2,3-epoxypropyltrimethylammonium chloride (26.58 grams) having the yellow color of 239 measured as Platinum-Cobalt Color, Test Method D 1209. Almost immediately, the Platinum-

Cobalt Color, Test Method D 1209, of the 2,3-epoxy-propyltrimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 120.

5

Example 5

The procedure of Example 4 is repeated except that 0.083 gram of sodium hypochlorite (providing about 406 ppm of active chlorine) is used. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyltrimethyl-
10 ammonium chloride aqueous solution thus obtained is reduced to a reading of 72.

Example 6

15

The procedure of Example 4 is repeated except that 0.261 gram of sodium hypochlorite (providing about 1277 ppm of active chlorine) is used. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyl-
20 trimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 39.

Example 7

25

The procedure of Example 4 is repeated except that 0.530 gram of sodium hypochlorite (providing about 2592 ppm of active chlorine) is used. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyl-
30 trimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 37.

Example 8

The procedure of Example 4 is repeated except that 0.820 gram of sodium hypochlorite (providing about 4011 ppm of active chlorine) is used. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyl-trimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 39.

10 Example 9

The procedure of Example 4 is repeated except that 1.108 grams of sodium hypochlorite (providing about 5419 ppm of active chlorine) is used. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyl-trimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 41.

20 Example 10

The procedure of Example 4 is repeated except that 1.529 grams of sodium hypochlorite (providing about 7478 ppm of active chlorine) is used. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyl-trimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 45.

Example 11

30 The procedure of Example 4 is repeated except that 1.934 grams of sodium hypochlorite (providing about 9459 ppm of active chlorine) is used. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyl-

trimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 64.

Example 12

5

The procedure of Example 4 is repeated except that 2.131 grams of sodium hypochlorite (providing about 10422 ppm of active chlorine) is used. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyl-
10 trimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 65.

Example 13

15

The procedure of Example 4 is repeated except that 2.390 grams of sodium hypochlorite (providing about 11689 ppm of active chlorine) is used. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyl-
trimethylammonium chloride aqueous solution thus obtained
20 is reduced to a reading of 90.

Example 14

The procedure of Example 4 is repeated except that
25 2.591 grams of sodium hypochlorite (providing about 12672 ppm of active chlorine) is used. The Platinum-Cobalt Color, Test Method D 1209, of the 2,3-epoxypropyl-
trimethylammonium chloride aqueous solution thus obtained is reduced to a reading of 125.

30

Example 15

An Erlenmeyer flask containing solution of sodium sulfate (12.5 g) and 25 percent by weight sodium

hydroxide(1.43 ml) in purified water (33 ml) is placed in a water bath kept at about 50 °C. While stirring the solution with a magnetic stirrer, corn starch (22.16 g) is added to the flask. The resulting starch slurry is
5 stirred for 10 minutes and then 2,3-epoxypropyltrimethyl-ammonium chloride obtained in Example 1 above is added to the starch slurry in an amount to provide a theoretical degree of substitution of 0.05. The reaction is allowed to proceed for 4 hours at a temperature of 50 °C. The pH
10 of the starch reaction slurry is lowered to pH=5-6 by the addition of 6N hydrochloric acid and then the resulting starch slurry is filtered through Whatman 113 filter paper under vacuum. The cationic starch thus obtained is washed with water (100 ml) three times and then dried in an oven
15 at about 50 °C. The starch was analyzed for nitrogen content. The analysis shows that it contained 0.4 percent nitrogen which is typical for these experimental conditions.

WHAT IS CLAIMED IS:

1. A process for eliminating or reducing color of an aqueous solution of 2,3-epoxypropyltrialkylammonium halide which process comprises treating an aqueous solution of colored 2,3-epoxypropyltrialkylammonium halide with an effective amount of an alkali metal salt of hypochlorous acid, hypochlorous acid or chlorine.
2. The process of Claim 1 wherein an alkali metal salt of hypochlorous acid, hypochlorous acid or aqueous chlorine is used in an amount to provide from 200 to 15,000 ppm of active chlorine.
3. The process of Claim 2 wherein an alkali metal salt of hypochlorous acid, hypochlorous acid or aqueous chlorine is used in an amount to provide from 600 to 2,500 ppm of active chlorine.
4. The process of any one of Claim 1 wherein 2,3-epoxypropyltrialkylammonium halide is treated with sodium hypochlorite.
5. The process of any one of Claims 1 to 4 wherein the aqueous solution of colored 2,3-epoxypropyltrialkylammonium halide is an aqueous solution of colored 2,3-epoxypropyltrimethylammonium chloride or an aqueous solution of colored 2,3-epoxypropyltrimethylammonium bromide.
6. The process of Claim 1 wherein the reduced color aqueous solution of 2,3-epoxypropyltrialkylammonium halide is further reacted with a substrate to produce a cationic substrate.

7. The process of Claim 6 wherein the substrate is starch.

5 8. A cationic substrate prepared by the steps of
(1) treating an aqueous solution of colored 2,3-epoxy-
propyltrialkylammonium halide with an effective amount of
an alkali metal salt of hypochlorous acid, hypochlorous
acid, or chlorine to reduce its color; and (2) reacting so
10 obtained aqueous solution of 2,3-epoxypropyltrialkyl-
ammonium halide with a substrate to form a cationic
substrate.

 9. The process of Claim 1 wherein the reduced color
15 aqueous solution of 2,3-epoxy-propyltrialkylammonium
halide is further reacted with a fiber to produce a
cationic fiber.

 10. A cationic fiber prepared by the steps of (1)
20 treating an aqueous solution of colored 2,3-epoxypropyl-
trialkylammonium halide with an effective amount of an
alkali metal salt of hypochlorous acid, hypochlorous acid,
or chlorine to reduce its color; and (2) reacting so
obtained aqueous solution of 2,3-epoxypropyl-
25 trialkylammonium halide with a fiber to form a cationic
fiber.

INTERNATIONAL SEARCH REPORT

International application No
PCT/US 99/18369

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C07D301/32 C08B31/12 D06M13/385

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C07D C08B D06M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X A	DE 20 55 046 A (HOFFMAN'S STÄRKEFABRIKEN AG) 18 May 1972 (1972-05-18) page 1 ---	8, 10 1-7, 9
X A	US 4 066 673 A (DOUGHTY JOSEPH B ET AL) 3 January 1978 (1978-01-03) column 1 ---	8 1-7, 9, 10
X A	US 5 006 125 A (PATTON ROBERT T ET AL) 9 April 1991 (1991-04-09) column 1 -----	8, 10 1-9

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

28 January 2000

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04/02/2000

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Steendijk, M

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 99/18369

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