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3,335,105

ANTISTATIC COMPOSITION COMPRISING THE REACTION PRODUCT OF A POLYEPOXIDE, A POLYAMINE, AND A NITROGEN CONTAINING COMPOUND

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This invention relates to synthetic fibers having thereon an antistatic finish which minimizes the accumulation of charges of static electricity during normal use and finishing operations. More particularly, this invention relates to treated synthetic fibers which process readily through the usual operations of textile manufacture without the application of special processing agents and which have a resistance to the accumulation of static electricity which is not lost after repeated washings.

Synthetic fibers are at a distinct disadvantage when compared with natural fibers in at least one respect for synthetic fibers have the tendency to collect static electrical charges during the manufacture and wearing thereof. Finished synthetic textile fabrics fail to drape like cotton or wool due to the accumulation of the static charges thereon and have a tendency to cling uncomfortably to the wearer. The static charges also attract dust and lint to the fabric. It is well known in the art that polyamines are effective temporary antistatic agents but lack durability when washed and have the tendency to impart undesirable characteristics to the fiber to which they are applied, such as, unpleasant hand, discoloration and the like. The problem of durability may be eliminated if high molecular weight polyamines are created by polymerization on the surface of the fibers from lower polyamines and dihalogen compounds, but this is not practical for the high temperature required adversely affects the fiber. Another more successful approach has been to cross-link the polyamines on the surface of the fiber by means of an epoxy resin. This approach has been successfully demonstrated when applied to the completed fabric and to a lesser degree when applied to a finished fiber. A more desirable approach would be to apply the antistatic coating to the fiber during the spinning thereof but this has not proved feasible with typical polyamine/epoxy compositions because of the adhesive characteristics of the epoxy group.

Amine/epoxy compositions are known to be effective adhesives and may be used for cementing together not only similar materials but such dissimilar materials as metals, wood, fabric, glass, and rubber. When a polyamine/epoxy composition of the prior art was applied in an aqueous suspension in a known manner to an acrylic fiber during the spinning operation, there was an extreme buildup of the polymerized polyamine/epoxy material on the curing roll surfaces. Individual filaments were cemented together in bundles and the cementing of some of the filaments to the roll surfaces resulted in many broken filaments and wholly unsatisfactory spinning results.

However, for some uses, the polyamine/epoxy composition may be padded on a finished fabric, film or any hydrophobic shaped structure. That substrate is then protected from the collection of excess static charges by the finish. Treatment of the finished fiber with the same polyamine/epoxy composition and curing either by heat treatment of prolonged standing of the fiber under ambient conditions in staple form avoided the problems associated with application during the spinning of the fiber, but gave a harsh fiber product which processed extremely poorly on woolen systems machinery and which

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showed high card loading that could not be corrected by the application of softeners, lubricants, or other finishing agents to the fiber.

Therefore, an object of this invention is to provide a novel process and composition of matter which will impart antistatic protection to a synthetic, hydrophobic shaped structure without causing objectionable changes in the properties thereof.

Another object of this invention is to provide a process which will continuously impart durable antistatic protection to a synthetic textile fiber during the manufacture thereof.

A further object of this invention is to provide a synthetic fiber having a durable antistatic finish which is acceptable to the woolen system for textile processing without further treatment.

These and other objects will become apparent in the course of the following specification and claims.

In accordance with a preferred embodiment of the present invention, a synthetic textile fiber is provided with durable antistatic properties by continuously associating the fiber during the spinning thereof with an aqueous polyamine/epoxy composition which may also contain a third ingredient. Where the fiber is formed from an acrylic polymer, the composition was applied before the fiber had completely collapsed, for upon the closing of the fissures in the fiber, a number of the polyamine/epoxy molecules are trapped therein thereby giving added durability to the finish. The polyamine/epoxy molecule carries a plus charge due to the presence of a quaternary ammonium group on the end thereof. This aids the oxygen and nitrogen atoms, which are present in the long-chain molecule, in the conduction of static electrical charges. The third ingredient mentioned prevents the filaments from sticking together during the spinning thereof and from adhering to the textile machinery. This ingredient is not a necessary one unless the finish is applied to the fibers before being processed on standard textile equipment. Therefore, the particular use of the antistatic composition determines whether the third ingredient is to be added to the finish composition.

The use of these antistatic agents, in accordance with the present invention, effects improvements in the characteristics of acrylonitrile polymers and articles produced therefrom. The invention is applicable not only to polyacrylonitrile, but also to copolymers, interpolymers, and blends thereof, particularly those containing at least 80 percent by weight of polymerized or copolymerized acrylonitrile. Such polymeric materials include acrylonitrile fiber-forming polymers with readily dyeable basic copolymers, the blend having an overall polymerized acrylonitrile content of at least 80 percent by weight.

For example, the polymer may be a copolymer of from 80 to 98 percent of acrylonitrile and from 2 to 20 percent of another copolymerizable mono-olefinic monomer. Suitable copolymerizable mono-olefinic monomers include acrylic, alphachloroacrylic and methacrylic acids, the acrylates, such as methylmethacrylate, ethylmethacrylate, butylmethacrylate, methoxymethyl methacrylate, beta-chloroethyl methacrylate, and the corresponding esters of acrylic and alpha-chloroacrylic acids; vinyl chloride, vinyl fluoride, vinyl bromide, vinylidene chloride, 1-chloro-1-bromoethylene; methacrylonitrile; acrylamide and methacrylamide; alpha-chloroacrylamide, or monoalkyl substitution products thereof; methyl vinyl ketone; vinyl carboxylates, such as vinyl acetate, vinyl chloroacetate, vinyl propionate, and vinyl stearate; N-vinyl-imides, such as N-vinylphthalimide and N-vinylsuccinimide; methylene malonic esters; itaconic acid and itaconic ester; N-vinyl carbazole; vinyl furan; alkyl vinyl esters; vinyl sulfonic acid; ethylene alpha,beta-dicarboxylic acids or their an-

hydrides or derivatives, such as diethylcitrate, diethylmesaconate; styrene; vinyl naphthalene; vinyl-substituted tertiary heterocyclic amines such as the vinylpyridines and alkyl-substituted vinylpyridines for example, 2-vinylpyridine, 4-vinylpyridine, 2-methyl-5-vinylpyridine, and the like; 1-vinylimidazole and alkyl-substituted 1-vinylimidazoles, such as 2-, 4-, or 5-methyl-1-vinylimidazole, vinylpyrrolidone, vinylpiperidone, and other mono-olefinic copolymerizable monomeric materials.

The polymer can be a ternary interpolymer for example, products obtained by the interpolymerization of acrylonitrile and two or more of any of the monomers, other than acrylonitrile, enumerated above. More specifically, and preferably, the ternary polymers contain from 80 to 98 percent of acrylonitrile, from 1 to 10 percent of a vinylpyridine or 1-vinylimidazole, and from 1 to 18 percent of another copolymerizable mono-olefinic substance, such as methacrylonitrile, vinyl acetate, methylmethacrylate, vinyl chloride, vinylidene chloride, and the like.

The polymer can also be a blend of polyacrylonitrile or a copolymer of from 90 to 99 percent acrylonitrile and from 1 to 20 percent of at least one other mono-olefinic copolymerizable monomeric substance with from 2 to 50 percent of the weight of the blend of a copolymer of from 30 to 90 percent of a vinyl substituted tertiary heterocyclic amine and from 10 to 70 percent of at least one other mono-olefinic copolymerizable monomer. Preferably, when the polymeric material comprises a blend, it will be a blend of from 80 to 99 percent of a copolymer of 80 to 98 percent acrylonitrile and from 2 to 20 percent of another mono-olefinic monomer, such as vinyl acetate, which is not receptive to dyestuff, with from 1 to 20 percent of a copolymer of from 30 to 90 percent of a vinyl-substituted tertiary heterocyclic amine, such as vinylpyridine, a 1-vinylimidazole, or a vinyl lactam, and from 10 to 70 percent of acrylonitrile to give a dyeable blend having an overall vinyl-substituted tertiary heterocyclic amine content of from 2 to 10 percent, based on the weight of the blend.

Such a fiber may be formed by extruding a typical acrylonitrile-based spinning dope through a conventional spinnerette into an aqueous coagulation bath wherein the dope hardens into a filament. After the fibers were washed and stretched as they were drawn up a hot water cascade, they were heated and partially dried on a set of drying rolls. When the moisture content of the fiber was from 10 to 50 percent by weight and the temperature of the filaments has reached approximately 100° C., they are immersed in an aqueous emulsion which contains:

- (a) A polyamine,
- (b) An epoxy resin, and
- (c) A third ingredient containing at least one uninterrupted hydrocarbon chain of at least 10 carbon atoms and at least one nitrogen atom capable of reaction with said polyepoxide.

These three ingredients are described in more detail hereinafter. The mixture preferably also contains a dispersing or emulsifying agent which insures a more homogeneous suspension of the several ingredients and, because of the reactive nature of these ingredients, it is preferably held at temperatures below about 40° C. in order to prevent premature polymerization and to thus extend the useful pot life of the mixture. The treated fibers then continue through a series of heated rolls during which time the fibers are completely dried and the antistatic coating is polymerized by the heat from the rolls thereby forming a veneer around the fibers. The time required for a segment of fiber to complete its course through the dual series of heated rolls and the application bath is typically 30 to 60 seconds. This is approximately the length of time required for treating the fiber with the antistatic emulsion and for making the same water insoluble. After the fiber has been dried, if desired, it may be crimped, relaxed

in a steam atmosphere, recripped, if desired, and cut to any desired staple length. The product is now ready for textile processing without the application of any further textile finishing ingredients. It is obvious that the above-described process is not followed where the finish is applied to a fabric.

In accordance with the present invention, these antistatic agents may also be used in combination with other textile filaments, such as polyester fibers and articles produced therefrom. A fiber was formed by extruding a typical polyester melt, such as polyethylene terephthalate, through a conventional spinnerette downwardly through a chimney where the melt was air quenched and was hardened into filaments. The fibers were converged and collected on a bobbin as undrawn yarn. A number of bobbins were then combined and a tow was formed by continuously pulling the yarn therefrom. The tow was heated to at least 40° C. by being passed under a hot water spray. The fibrous tow then passed into a draw zone where the temperature thereof was increased to 80° C.-90° C. by another hot water spray or by a finishing solution and was elongated from four to five times its original length. After the tow has been drawn, it is immersed in or sprayed with an aqueous antistatic emulsion which becomes polymerized thereon when heated to about 145° C. The dried fibrous tow was then cut to staple lengths and processed according to conventional techniques.

While this invention has been generally directed to its use on acrylic fibers, the antistatic finish is especially useful on fibers composed of polyamide fibers. Simple linear polyamides which are normally insoluble in alcohol but are soluble in phenols and they are of two types, those derived from polymerizable monoaminomonocarboxylic acids or their amide forming derivatives and those derived from the reaction of a suitable diamine with suitable dicarboxylic acids. On hydrolysis with mineral acids, the polyamides yield monomeric amide forming reactants. The activating compounds of this invention are applicable to the polycarbonamides of the type derived from the reaction of a suitable diamine with a suitable dicarboxylic acid. On hydrolysis with hydrochloric acid, these polycarbonamides yield the dibasic acid and the diamine hydrochloride. These polycarbonamides are of the general type disclosed in U.S. Patents 2,071,250, 2,071,253, and 3,130,948. The term polycarbonamide means polymers having recurring units of formula:



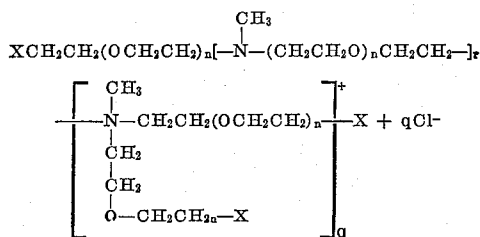
- 50 where R is hydrogen or a monovalent hydrocarbon radical as integral parts of the main polymer chain, the average number of carbon atoms separating the amide groups being at least two.

In particular, this invention is concerned with the simple, unsubstituted polyamides such as the polymers formed by the reaction of tetramethylene diamine and adipic acid, tetramethylenediamine suberic acid, tetramethylenediamine with sebacic acid, hexamethylenediamine with adipic acid, hexamethylenediamine with suberic acid and hexamethylenediamine with sebacic acid. Broadly, this invention will cover any nylon formed from a diamine and a dicarboxylic acid as well as those formed from the interpolymerization of monoaminomonocarboxylic acid. In addition, polymers formed from the reaction of two or more diamines with dicarboxylic acids and/or two or more dicarboxylic acids with diamines are contemplated. Thus, the word polyamide is defined as above for the purpose of this invention and when referred to in the specification encompasses the above monoaminomonocarboxylic acid polyamide as well as the diamine-diacid polyamides.

The permanency of the antistatic coating which has been applied to the fibers was checked in the following manner. The fibers are converted into yarn and the yarn is knitted into a tubular fabric shape whereupon the fabric

is blank dyed by any conventional dyeing process. In the examples below, it will be seen that all of the samples were not blank dyed. The fabric is then washed in a Kenmore washing machine at 110° F. using 0.5 percent aqueous solution "Tide" detergent for four minutes or alternatively, a 0.3 percent solution of tetra sodium pyrophosphate may be used as the detergent. After the tube is removed from the washing machine, it is centrifuged to remove the excess moisture. The steps of washing the tube in the detergent for four minutes and centrifuging it are repeated four times whereupon it is dried in an air atmosphere with a relative humidity of 35 percent at a temperature of 72° F. The tube is then run on a Hayeck and Chromey apparatus to determine the level of static protection. The protection is considered satisfactory if one-half of the static electrical charge induced thereon is drained off within 60 seconds. After 15 washings of fabric samples embodying the present invention, each of which being conducted in the above-described manner, the half-life was less than 60 seconds thereby indicating that the antistatic coating was permanently applied thereto.

An example of a polyamine in accordance with the present invention is a polyamine of the formula:

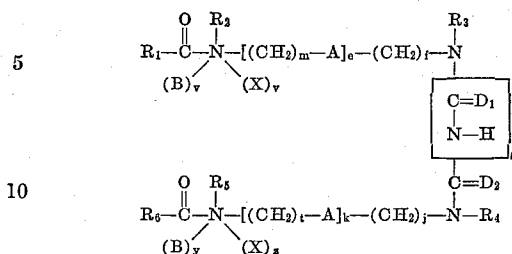


wherein n is a whole integer from 2 to 40, X is selected from the group consisting of Cl and NHCH_3 , r is a whole number from about 30 to about 100 and q is a whole number from about 4 to about 14. Where the above structure contains a covalent chlorine end group, it is contemplated that it could react further with a secondary or tertiary amine group to form a still more complex structure. The preparation of this class of polyamines is discussed in U.S. Patent No. 3,070,552 to Tesoro, granted December 25, 1962. In general, polyethylene glycol 600 is reacted with thionyl chloride to form polyethylene glycol 600 dichloride. The latter is extended with monoethylamine so that both active hydrogens react and two molecules of HCl are split out. The final mechanism for cross-linking the finish to render it permanent by reaction with an epoxide is assumed to proceed through the hydrogen of the secondary amine. Such amines may be methylamine, ethylamine, propylamine, butylamine, amylamine, hexylamine, dodecylamine, 3-isopropyl-n-propamine and the like. Such polyamines may be purchased on the open market from the Onyx Chemical Company of 190 Warren Street, Jersey City, New Jersey, under the trademark "Aston 1022."

The epoxide referred to above can be prepared by condensing epichlorohydrin with a polyol. A typical example is the condensation product (with elimination of HCl) of glycerine and epichlorohydrin. Preparation of these products is described in detail in British Patent 780,288, dated July 31, 1957. Such a material with a molecular weight of between about 230 to 360 is available on the open market under the trademark "Eponite 100" (viscosity 90-150 cp. at 25° C.; 10.2 lb./gal.) by Shell Chemical Corporation of 500 Fifth Avenue, New York, New York. Other similar commercial products from the same source include the "Epon" resins, which are diepoxide condensation products of epichlorohydrin and 2,2 diphenylol propane, and the poly(allyl glycidyl ethers).

The third critical compound which is to be used when the finish is to be applied to the fiber during spinning or before the textile processing thereof may be substituted carbamyl compounds being formed from a condensation product, such as, substituted ureas, thioureas, biurets,

guanidines, diguanidines, or guanyl ureas of high molecular weight of the following general formula:



In this formula, R_1 corresponds preferably to a saturated aliphatic chain containing six or more carbons, although it may also represent an unsaturated hydrocarbon, or it may carry other substituting groups, such as hydroxyl, carboxyl, or chlorine. In each case, it may be used as a source to introduce this radical into the condensation products as will be shown later. R_6 is used to indicate hydrocarbon groups of similar type to R_1 and in a given compound may be identical with R_1 or be secured from a fatty acid.

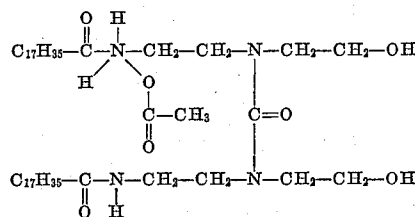
In the formula R_2 , R_3 , R_4 , and R_5 may represent either hydrogen, a simple alkyl of less than five carbons, or a similar hydroxy alkyl radical such as a beta ethanol group. R_3 and R_4 may also represent alternatively an acid amide chain derived from an acid and a polyamine.

The chemical groups indicated by D_1 and D_2 may be oxygen ($=\text{O}$) in the case of the ureas, sulphur ($=\text{S}$) with the thioureas, or imido ($=\text{NH}$) with the guanidines. The letter g represents a small whole number ranging from 0 to 3 and while D_1 and D_2 may be the same as in the biurets they may be different as in the guanyl ureas.

Depending upon the nature of the aliphatic polyamine used in the condensation with the acids, the letter A is used to represent either an amino $-(\text{NH})-$ group, a simple alkyl or alkylol substituted amino of less than five carbons, such as, $-(\text{NC}_2\text{H}_4\text{OH})-$ or $-(\text{NC}_2\text{H}_5)-$, or oxygen, or sulphur. The letters f , i , e , k , m , and t represent small integers, f and j varying from 1 to 6, e and k from 0 to 6, m and t from 1 to 6, and any of the hydrogens in the CH_2 groups may be substituted by a simple alkyl or a hydroxy group.

The condensation products, as identified above, used are in many cases directly water soluble or water dispersible. In all cases, the condensations, as identified above, are readily dissolved after treatment with acids, such as, acetic, lactic, boric, oxalic, benzoic, salicylic, furoic, citric, tartaric, formic, phthalic, succinic, or alkyl naphthalene sulphonic acids or after reaction with an alkylating agent, such as, ethyl chloride, benzyl chloride, ethylene oxide, ethylene chlorohydrin, or dimethyl sulphate. The salts are alkylated products of our substituted urea condensates are indicated in the general formula given where B represents the acid hydrogen or alkyl groups and X the acid radical or halogen group, while for primary urea, etc., condensations B and X disappear from the formula. Thus, u , v , y and z are whole integers varying from 0 to 1.

In reference to the above-mentioned general formula for the substituted carbamyl compounds, the compound of the specific chemical formula as shown below was added in combination with the polyamine/polyepoxide mixture:

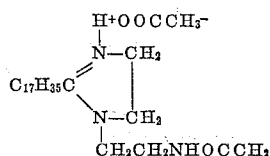


wherein the above chemical formula has the chemical name of α,β -dihydroxyethyl- α,β -di(beta stearmidoethyl) urea monoacetate. The preparation of the above is discussed in U.S. Patent 2,304,113, to Morgan et al. and can be purchased on the open market from Arnold Hoffman and Company of 55 Canal Street, Providence, Rhode Island, under the trademark "Ahcovel A." It has been found that this compound enters into the polymerization scheme with the polyamine/polypoxide composition and becomes cross-linked therewith. By cross-linking the "Ahcovel A" (as identified above) molecule with the polyamine/polypoxide molecule, a new composition of matter results which has all of the antistatic advantages of the polyamine/polypoxide reaction product plus new advantages which will be pointed out. This compound serves the main functions of acting as a conductor of electrons which have accumulated on the surface of the fiber and of providing a lubricant for the filaments so as to make the mechanical flow thereof through the processing systems proceed smoothly. Also, the filaments when cut to staple lengths behave in a manner like natural fibers when processed on the cotton system and have a relatively low card loading. Where only the polyamine/polypoxide reaction product is used as an antistatic agent, it can only be applied to the fibers within any degree of success after they have been woven or knitted into a fabric, otherwise, the disadvantages that have previously been pointed out result.

The third critical compound may also be an alkyl substituted imidazoline derivative of the type described in U.S. Patent No. 2,200,815, to Ackley which may be obtained on the open market from Onyx Chemical and Oil Company of 190 Warren Street, Jersey City, New Jersey, under the trademark "Onyxsan." Compounds of this type may be prepared by acylating a primary or secondary amino group of a μ -alkyl N-amino alkyl imidazoline. Such acylation may take place either prior to or during neutralization, with an organic or inorganic acid, of the tertiary amino group of the μ -alkyl N-amino alkyl imidazoline. Generally the acylation and neutralization are performed simultaneously, although, under proper conditions, the steps of acylation and neutralization may be carried out separately and in succession. The molecule is acylated by replacing a hydrogen atom which is attached to a nitrogen atom by the group



wherein Y is an alkyl, aryl or aralkyl group. When μ -heptadecyl N-amino ethyl imidazoline is neutralized with acetic anhydride, the following chemical formula appears to result:



This molecule also cross-links with the polyamine/polypoxide composition and when polymerized on the surface of a filament, a new composition of matter results which has the antistatic advantages as outlined above.

A dispersing agent is preferably added to the aqueous bath to insure a thorough mixture of the respective ingredients. Satisfactory dispersing agents are the aromatic polyglycol ethers, one example of such being the alkylphenol polyglycol ether containing 9.5 moles of ethylene oxide which can be purchased on the open market under the trademark "Neutronyx 600" and which is manufactured by the Onyx Chemical Corporation, 190 Warren Street, Jersey City, New Jersey. Another satisfactory dispersing agent is nonylphenol polyethylene glycol ether which can be purchased on the open market under the trade name "Tergitol" and which is manufactured by Union Carbide Corporation, 270 Park Avenue, New York, New York.

The following examples are cited to illustrate the invention. They are not intended to limit it in any way.

Example I

Acrylic fibers containing a copolymer of 93 percent acrylonitrile and 7 percent vinyl acetate were wet spun in a conventional manner. Prior to the final drying the coagulated, washed and partially collapsed fiber, it was passed through a bath comprising an aqueous emulsion as described below, the concentration thereof being adjusted to afford a solids pickup of 0.70 based on the weight of the fiber. The pH of the emulsion was reduced to 7.0 by phosphoric acid.

An aqueous emulsion was prepared by making the concentration of the final solution, the percentages of which are based on the absolute weight of the constituents, as follows:

	Percent
Aston	5.00
Ahcovel A	1.33
Eponite 100	0.63
Neutronyx 600	0.14
Water	92.90

After being passed through the bath, the fibers were dried on steam heated rolls, treated momentarily with steam at 35 p.s.i. superatmosphere pressure, cooled to ambient conditions and cut to staple lengths.

Several tests were run wherein the total finish applied to the fiber was varied over a wide range by changing the relative concentrations of the finish ingredients which comprise the aqueous bath. Table I (immediately below) shows that for each concentration of the ingredients, the static half-life varies and that after each textile process (that is, scouring blank dyeing and washing) the static half-life increases.

TABLE I

Sample	Treatment, percent Solution	pH	Static Half-Life in Seconds					
			Scoured	Scoured and Blank Dyed	Scoured and Blank Dyed Plus 2 Washes	Scoured and Blank Dyed Plus 5 Washes	Scoured and Blank Dyed Plus 7 Washes	Scoured and Blank Dyed Plus 10 Washes
A	5% Aston 1022 1.33% Ahcovel A 0.63% Eponite 100 0.14% Neutronyx	9.5	2	3	3	4	5	6
B	Adjust pH of A with H ₃ PO ₄	8.0	2	2	4	4	4	5
C	do	7.0	2	2	3	3	6	8
D	2.5% Aston 1022 0.66% Ahcovel A 0.32% Eponite 100 0.07% Neutronyx	9.3	2	3	4	8	8	9
E	Adjust pH of D with H ₃ PO ₄	8.0	3	3	4	6	6	9
F	do	7.0	3	4	7	9	9	10

Where the amount of finish is increased to a significant degree, the proper textile processing cannot be maintained; however, for the concentration shown above, this is no problem. The ideal is to give to the fiber the maximum amount of static properties without interfering with the textile processing characteristics thereof.

Example II

An acrylic fiber containing a copolymer of 93 percent acrylonitrile and 7 percent vinyl acetate was wet spun, stretched, dried and collected in a conventional manner. These fibers were then knitted into a tube which was dipped into an emulsion. The emulsion consisted of:

	Percent
Aston 1022	5.00
Eponite 100	0.50
Neutronyx 600	0.125
Water	94.375

The fibers were dried and cured for 10 minutes at 280° F. After being conditioned overnight at 72° F. and 33 percent RH, the treated swatches were evaluated for its

though there was no similarity in the fibers used for polyester fibers were used in this example.

Example IV

Polyethylene terephthalate fibers were spun in the conventional manner. The aqueous emulsion as set forth below was prepared in bath form and the fibers, after having been spun and knitted into tubing, are run there-through. These fibers are dried and cured for 10 minutes at 290° F. Nylon was spun in the conventional manner emersed in the emulsion as set forth below, dried and cured for 10 minutes at 290° F.

An aqueous emulsion was prepared by making the concentration of the final solution, the percentages of which are based on the absolute weight of constituents, as follows:

	Percent
Aston 1022	5.0
Ahcoval A	0.40
Eponite 100	0.50
Neutronyx 600	0.125

The above emulsion was diluted one to one with water and was applied to the fiber as set forth above. The results are set forth in Table III.

TABLE III

Fabric	Treatment, Percent Solution	Static Half-Life in Seconds		
		Scoured and Blank Dyed Plus 5 Washes	Scoured and Blank Dyed Plus 10 Washes	Scoured and Blank Dyed Plus 20 Washes
Polyester Nylon.....	5% Aston 1022..... 0.50% Eponite 100..... 0.40% Ahcoval A..... 0.125% Neutronyx 600.....	0 2	10 4	20 5
Polyester Nylon.....	2.5% Aston 1022..... 0.25% Eponite 100..... 0.20% Ahcoval A..... 0.0625% Neutronyx 600.....	0 2	14 13	31 15

static collecting propensities on a Hayeck-Chromey apparatus.

TABLE II

Fabric	Treatment, Percent Solution	Static Half-Life in Seconds				
		Scoured and Blank Dyed Plus 5 washes	Scoured and Blank Dyed Plus 10 washes	Scoured and Blank Dyed Plus 15 washes	Scoured and Blank Dyed Plus 20 washes	Scoured and Blank Dyed Plus 30 washes
Acrilan.....	5% Aston 1022..... 0.5% Eponite 100..... 0.125% Neutronyx 600.....	3	5	8	12	21
Acrilan.....	2.5% Aston 1022..... 0.25% Eponite 100..... 0.063% Neutronyx 600.....	3	6	9	14	34
Nylon.....	2.5% Aston 1022..... 0.25% Eponite 100..... 0.08% Neutronyx 600.....	2	3	3	4	7

Example III

A tube was knitted from a polyethylene terephthalate yarn and was dipped into an emulsion prepared as shown in Example II. After being dipped or padded in the emulsion, the tube was cured as taught in Example II. The emulsion consisted of:

	Percent
Aston 1022	5.00
Eponite 100	0.50
Neutronyx 600	0.125
Water	94.275

The results showed that after 5 washes the static half-life was 15 seconds and after 15 washes the static half-life was 31 seconds. In this example, the tube was blank dyed before it was emersed in the emulsion. In Example II, the tubes were not blank dyed before being padded and on the whole, the antistatic properties were better al-

Example V

Polyethylene terephthalate fibers were spun in the conventional manner. The aqueous emulsion as set forth below was prepared in bath form and the fibers, after having been spun and knitted into tubing, are run there-through. These fibers are dried and cured for 10 minutes at 290° F. Nylon was spun in the conventional manner emersed in the emulsion as set forth below, dried and cured for 10 minutes at 290° F.

An aqueous emulsion was prepared by making the concentration of the final solution, the percentages of which are based on the absolute weight of constituents, as follows:

	Percent
Aston 1022	5.0
Eponite 100	0.5
Neutronyx 600	0.125

TABLE IV

Fabric	Treatment, Percent Solution	Static Half-Life in Seconds		
		Scoured and Blank Dyed Plus 5 Washes	Scoured and Blank Dyed Plus 10 Washes	Scoured and Blank Dyed Plus 20 Washes
Polyester Nylon	5% Aston 1022	0	9	15
	0.5% Eponite 100	0	2	2
	0.125% Neutronyx 600	0	13	24
Polyester Nylon	2.5% Aston 1022	2	5	7
	0.25% Eponite 100			
	0.0625% Neutronyx 600			

Example VI

The acrylic fibers as described in Example I and the polyethylene terephthalate and nylon fibers as described in Example IV were emersed in the aqueous emulsion as set forth below. These fibers were then dried and cured for 10 minutes at 290° F.

The aqueous emulsion was prepared by making the concentration of the final solution, the percentages of which are based on the absolute weight of constituents, as follows:

	Percent
Aston 1022	5.0
Eponite 100	0.5
Onyxsan W	0.40
Neutronyx 600	0.125

TABLE V

Fabric	Static Half-Life in Seconds		
	Scoured and Blank Dyed Plus 5 washes	Scoured and Blank Dyed Plus 10 Washes	Scoured and Blank Dyed Plus 20 Washes
Acrylic	2	10	24
Polyester	0	9	19
Nylon	2	3	5

As previously described, a hydrophobic shaped structure and, more particularly, a synthetic textile fiber or fabric is treated with an aqueous emulsion comprising a mixture of a polyepoxide, a polyamine and, alternatively, a compound containing at least one uninterrupted hydrocarbon chain of at least 10 carbon atoms and at least one nitrogen atom capable of reaction with the polyepoxide may be polymerized therewith.

This invention discloses the cross-linking of a polyepoxide with a new polyamine for use on a processed textile fabric or any synthetic, hydrophobic material to render the same static free. The versatility of this composition is obvious for it may further receive a third component, in the polymerization scheme. The long chain molecules of this third component link to the polyepoxide and extends outwardly from the surface of the fiber thereby forming tentacles which prevent foreign objects from coming into touch with the polyepoxide and from adhering to the same. This enables the manufacturer to apply the antistatic finish either during the spinning process or after the textile processing thereof.

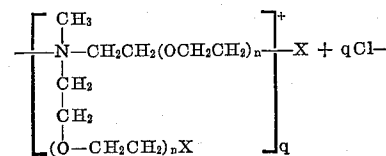
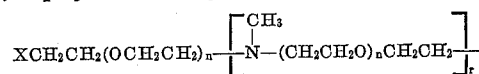
Many different modifications of the invention may be made without departing from the scope and spirit thereof. It is contemplated that variations may be made in the percentages of the compound used without greatly altering the antistatic characteristics of the fiber; therefore, the applicant does not wish to be bound by the numbers exactly as they appear above.

We claim:

1. A composition suitable for minimizing the accumulation of charges of static electricity on synthetic textile

fibers, said composition comprising the condensation product of:

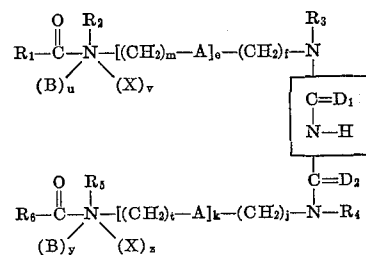
- (A) a polyepoxide;
(B) a polyamine having the formula



wherein X is selected from the groups consisting of —Cl and —NHCH₃, n is a whole integer from about 2 to about 40, r is a whole integer from about 30 to about 100 and q is a whole integer from about 4 to about 14; and

- (C) a nitrogen containing compound selected from the group consisting of

- (1) a substituted carbamyl compound having the formula:



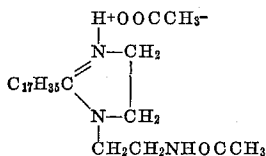
wherein R₁ and R₆ are hydrocarbon radicals containing at least about 6 carbon atoms, R₂ and R₅ are selected from the group consisting of hydrogen, an alkyl radical containing up to about 5 carbon atoms and a hydroxy alkyl radical containing up to 5 carbon atoms, R₃ and R₄ are selected from the group consisting of hydrogen, an alkyl radical containing up to about 5 carbon atoms, a hydroxy alkyl radical containing up to about 5 carbon atoms and an acid amide chain derived from an organic acid and a polyamine, B is selected from the group consisting of hydrogen and alkyl, X is selected from the group consisting of an organic acid radical and a halogen, u, v, y and z are whole integers varying from 0 to 1, A is selected from the group consisting of amino, oxygen, sulphur, an alkyl containing up to about 5 carbon atoms and an alkylol substituted amino containing up to about 5 carbon atoms, f, j, m and t are whole integers varying from about 1 to about 6, and e and l are whole integers varying from 0 to about 6; and

- (2) an alkyl substituted imidazoline compound prepared by acylating a primary or secondary amino group of a μ-alkyl N-amino alkyl imidazoline.

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2. The composition of claim 1 wherein the polyepoxide is a reaction product of glycerin and epichlorohydrin.

3. The composition of claim 1 wherein the nitrogen containing compound is an alkyl substituted imidazoline compound having the formula



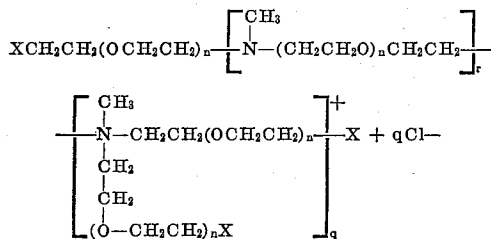
4. The composition of claim 1 in the form of an aqueous emulsion.

5. A synthetic textile bearing the composition of claim 1.

6. A composition suitable for minimizing the accumulation of charges of static electricity on synthetic textile fibers, said composition comprising the condensation product of

(A) a polyepoxide;

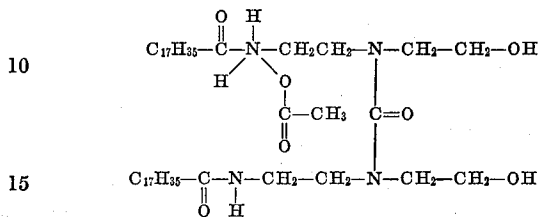
(B) a polyamine having the formula:



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wherein X is selected from the groups consisting of $-\text{Cl}$ and $-\text{NHCH}_3$, n is a whole integer from about 2 to about 40, r is a whole integer from about 30 to about 100 and q is a whole integer from about 4 to about 14; and

(C) a nitrogen containing compound having the formula



7. The composition of claim 6 in the form of an aqueous emulsion.

8. A synthetic textile bearing the composition of claim 6.

References Cited

UNITED STATES PATENTS

25	2,200,815	5/1940	Ackley	252-8.8
	2,304,113	12/1942	Morgan et al.	260-561
	2,730,464	1/1956	Winsor	252-8.8
	2,982,751	5/1961	Anthes	260-29.2
	3,070,552	12/1962	Tesoro et al.	260-29.2
30	3,175,987	5/1965	Pretka	260-29.2

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