United States Patent Office 3,716,393 Patented Feb. 13, 1973

3,716,393

PROCESS FOR ANTISTATICALLY TREATING ARTICLES

Robert L. Baker and Edward Szlosberg, Greenville, S.C., assignors to Phillips Fibers Corporation, Bartlesville, Okla.

Filed Feb. 8, 1971, Ser. No. 113,618
Int. Cl. B44d 1/092
U.S. Cl. 117—47 R 5 Claims

ABSTRACT OF THE DISCLOSURE

A process for treating an electrically chargeable article with an anionic resist-agent and thereafter treating the article with a cationic agent to condition said article for lower accumulation of static charges of electricity thereon.

This invention resides in a process for treating electrically chargeable articles with an antistatic agent. In another aspect, this invention resides in a process for more permanently affixing an antistatic agent to an electrically chargeable article.

Heretofore, articles have been treated with antistatic agents in order to condition said articles for lower accumulation of static charges of electricity thereon. These articles generally lose this antistatic conditioning in a short period of time particularly where the treated article is a material that is repeatedly laundered or cleaned.

It is therefore an object of this invention to provide a process for treating electrically chargeable articles with an antistatic agent that will lower the accumulation of static charges of electricity on said article for a longer period of time.

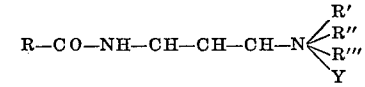
By the expression "electrically chargeable articles," as herein utilized, are meant molded articles, fabrics, filaments, films, yarns, fibers, and the like, that are formed of a material that can accumulate static charges of electricity. Examples of these materials, among others, are vinyl resins, thermoplastic vinyl resins, and high molecular artificial products which arise from organic bifunctional amino-, hydroxy-, carboxyl-, amino-hydroxy-, and carbonyl compounds by amide-like linkages which have also been called synthetic protein fibers, linear-polymeric amides, super polyamides, super polyurethanes, or synthetic polyamides and which today are known in the textile industry under registered names such as "nylon" or "perlon."

In the process of this invention, these articles, hereafter referred to as fibers for convenience, are first treated with an anionic resist-agent, as known in the art. One example of the anionic resist-agent treatment is to place the fibers in a volume of water and maintain the fibers in said water until said fibers are thoroughly wet out. The volume of anionic resist-agent, formed by condensing a mixture of naphthalene monosulfonic acid, dihydroxydiphenyl sulfone and formaldehyde is mixed with the water to form a resultant aqueous solution having about 0.1 to about 5 percent by weight anionic resist-agent. Another anionic resist-agent that can be utilized in the method of this invention are the sulfomethylated condensation products set forth in U.S. Pat. 3,322,488. The temperature of the anionic resist-agent solution having the fibers submerged therein is increased to and maintained at about 150–200° F. for a period of about 10 to about 120 minutes. It has been found that treatment of the fibers for a shorter period of time and/or at a lower temperature does not condition the fibers for retention of a satisfactory amount of the antistatic agent for an increased period of time. Treating of the fibers for longer periods of time than about 120 minutes does not appreciably increase the receptivity of the fibers and results in a waste

of labor, equipment and time. Higher temperature treatments can damage the material and also result in a waste of labor, equipment and time. Where fibers, materials, yarns, and other textiles are being treated, it has been found that the fibers can be more easily dyed and electrostatically treated if the fibers are dyed prior to subjecting the fibers to the antistatic treating process of this invention. In order to uniformly treat the fabric by the process of this invention, it is preferred that the anionic resist-agent bath have means disposed therein to agitate and move the fibers and/or aqueous solution during said treatment.

The fibers are then recovered from the anionic resist-agent bath and thereafter are thoroughly dried, preferably water rinsed and dried. The fibers are thereafter contacted with a cationic antistatic agent. The cationic antistatic agent can be brought into contact with the fibers by spraying said agent onto the fibers, dipping said fibers, spraying and exhausting, and by other methods known in the art. An effective method for treating the fibers with a cationic antistatic agent has been to spray said agent onto one surface of the fibers while vacuum means adjacent an opposed surface of the fibers moves the agent through said fibers.

The cationic antistatic agent used in this invention can be, for example, an aqueous solution of quaternary ammonium compounds, an aqueous emulsion of a cationic secondary fatty acid amide or a cationic amido-ester, as known in the art of cationic antistatic agents, for example, having a general formula of [RN]+X— where four carbon atoms are directly linked to the nitrogen atom of the compound through covalent lengths and an anionic is linked to the nitrogen through an electrovalent bond. More specifically, the general formula can be written as follows:



where R represents an aliphatic hydrocarbon radical containing at least 7 carbon atoms, R′ and R″ each represents an alkyl radical containing from 1 to 5 carbon atoms, inclusive, R‴ represents a radical of the group consisting of alkyl, alkenyl and aralkyl radicals, and Y represents an anion.

Illustrative examples of radicals represented by R in the formula are heptyl, octyl, octenyl, nonyl, nonenyl, decyl, decenyl, undecyl, undecenyl, tridecyl, tetradecyl, tetradecenyl, heptadecyl, heptadecenyl, octadecyl, octadecenyl, etc. Illustrative examples of radicals represented by R′ and R″ are methyl, ethyl, propyl, isopropyl, n-butyl, isobutyl, sec.-butyl, tert.-butyl, n-amyl, isoamyl, sec.-amyl, tert.-amyl and the other isomeric amyl radicals. Illustrative examples of radicals represented by R‴ are the same alkyl radicals as those just given with reference to R′ and R″ and, in addition, the higher alkyl radicals, e.g., hexyl to octadecyl, inclusive, the same alkenyl radicals as those given above with reference to R and, also, allyl, methallyl, ethallyl, 2-butenyl, 3-butenyl, 3-methyl-2-butenyl, 3-pentenyl, 4-pentenyl, 2,3-pentadienyl, 3-methyl-3-butenyl and the higher alkenyl radicals, e.g., hexenyl to octadecenyl, inclusive; and the various aralkyl radicals, e.g., benzyl, phenylethyl, phenylpropyl, phenylisopropyl, phenylbutyl, etc. Illustrative examples of anions represented by Y are the halide ions (that is, Y can represent a chloride, bromide, fluoride or iodide), sulfate, sulfonate, phosphate, hydroxide, borate, cyanide, carbonate, hydrocarbonate, thiocyanate, thiosulfate, isocyanate, sulfite, bisulfite, nitrate, nitrite, oxalate, silicate, sulfide, cyanate, acetate and the other common inorganic and organic anions.

The amount of the aforesaid liquid or liquid composition containing antistatic agent compound which is ap-

plied to the vinyl-resin article is such that the finished article has associated therewith from, by weight, 0.2% to 10% of the said amido compound, based on the weight of the dried, untreated article. The resulting article is then preferably dried by any suitable means. The antistatic treatment is applicable to filaments, fibers, yarns, films, woven, knitted and felted fabrics, and other articles made from or containing one or more thermoplastic vinyl resins for the purpose of eliminating substantially completely (in some cases) or materially lessening or retarding (in all other cases) the tendency of such articles to accumulate charges of static electricity thereon either during the production of the article, or in connection with subsequent finishing operations, or during the use of the article.

The antistatic agent is preferably mixed with water to form a 0.5% to about 5% by weight cationic antistatic solution. Examples of other volatile liquids with which the compound can be mixed to form the cationic solution used in this invention are, for example, ethyl alcohol, propyl alcohol, mixtures of water and alcohol, or any other volatile liquid solvent or dispersant medium which will not attack or otherwise detrimentally affect the fibers which are treated. Water is the preferred liquid medium for reasons of availability, economics, and ease in handling.

By so pretreating articles with an anionic resist-agent and treating the article with a cationic agent, the article is conditioned to resist the accumulation of electrical charges for longer periods of time. In order to test the retentivity of the antistatic properties of an article treated by the process of this invention, control tests were run on a nylon dyed carpet. The carpet was run in a water-filled paddle machine until the carpet was wet out. A 2% anionic resist-agent water solution was formed and the carpet was run therein for one hour at 175° F. Thereafter the carpet was extracted and dried at 225–250° F. A 2–4% aqueous cationic solution was brought into contact with the carpet for 20 minutes and thereafter the carpet was dried.

The carpet treated by this invention was thereafter conditioned for 24 hours at a humidity of about 46–50 percent. Carpet samples were thereafter repeatedly washed and tested by the "scuffing" body contact method on carpet samples treated by this invention, carpet samples untreated, and carpet samples that were only treated with an antistatic agent. In this method, the electrodes of an Atlab tester were held by a standing person. The person then made 10 swipes across the carpet with his shoe and the highest reading was recorded. This was repeated three times with each carpet sample and the average of the three readings was utilized for report purposes. Commercially available anionic resist agents and cationic agents were utilized. The anionic agent utilized was a product manufactured by Ciba-Geigy Corporation and marketed under the trade name of Erional NW (the condensation product as described above). The cationic agents utilized were by trade name and manufacturers, Alubrasol MM, a secondary fatty acid amide—Jordan Chemical Company, Synolube C and Synolube E—

Charles S. Tanner. As shown in the following table, dyed samples treated by the method of this invention are compared to sample tests on like carpet treated only with antistatic agent and also on untreated dyed carpet. All samples were conditioned overnight after laundering and before testing. The testing results are as follows:

BODY CONTACT TEST METHOD

[Static electricity, volts]

Sample identification	Initial	Number of launderings				
		1	2	3	4	5
Untreated	2,000	[2] 2,000	[2] 2,000	[2] 2,000	[2] 2,000	[2] 2,000
Alubrasol MM only	0	433	500	1,000	1,400	1,800
Alubrasol MM [1]	0	300	530	0	400	400
Synolube C	0	600	600	1,800	[2] 2,000	[2] 2,000
Do. [1]	0	100	160	0	400	500
Synolube E	0	500	500	400	900	1,100
Do. [1]	100	400	200	600	300	200

[1] Denotes sample treated by the process of this invention.
[2] Plus.

These tests show that by treating fibers by the method of this invention the durability of the antistatic properties is increased as indicated by the lower static electricity charges obtained after repeated launderings.

What is claimed is:

1. A process for treating electrically chargeable articles comprising:

contacting the article with an aqueous solution of about 0.1 percent to about 5.0 percent by weight of an anionic resist agent, said anionic resist agent being formed by condensing a mixture of naphthalene monosulfonic acid, dihydroxydiphenyl sulfone and formaldehyde; and

thereafter contacting the article with a cationic antistatic agent said cationic antistatic agent being one of an aqueous solution of secondary fatty acid amide or a cationic amido-ester.

2. A process, as set forth in claim **1**, wherein the article to be treated is dyed fibers.

3. A process, as set forth in claim **2**, wherein the fibers are treated in an anionic resist-agent bath at a temperature in the range of about 150° F. to about 200° F. and for a time of about 10 to about 120 minutes.

4. A process, as set forth in claim **2**, wherein the fibers are treated by spraying the antistatic agent onto said fibers.

5. A process, as set forth in claim **2**, wherein the cationic antistatic agent is sprayed onto one surface of the fibers while vacuum means adjacent an opposed surface of the fibers moves the agent through the fibers.

References Cited

UNITED STATES PATENTS

2,342,643 2/1944 Cessna ---------- 117—119 X

WILLIAM D. MARTIN, Primary Examiner

T. G. DAVIS, Assistant Examiner

U.S. Cl. X.R.

117—47 A, 104 R, 119, 138.8 D, 138.8 N, 138.8 UA