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2,985,543

**ANTI-STATIC TREATMENT AND PRODUCTS
RESULTING THEREFROM**

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8 Claims. (Cl. 117—139.5)

This invention relates to a method of treating textile materials. More particularly this invention relates to a method of treating synthetic textile materials for the purpose, among others, of providing a durable anti-static finish on such materials and to the treated textile materials obtained thereby.

One disadvantage of the new textile fibers and fabrics prepared from the recently introduced synthetic hydrophobic materials and of all textile fibers treated with conventional finishes is that they tend to develop a static electrical charge. This charge is objectionable during the manufacture of the textile and in the finished garment. During manufacture the static charge on the fiber or fabric prepared therefrom interferes with their convenient handling during treatment with various pieces of equipment such as spinning, reeling, weaving and the like. Finished articles which are designed to drape like cotton or wool articles fail to do this properly in that the fabrics often cling to the wearer. Furthermore, such textiles tend to collect dust and lint and the electric discharge itself is bothersome and may be extremely dangerous, for example, if occurring near flammable solvents as in a hospital operating room.

Many treating agents such as mono-, tri- and poly-ethanolamine salts have been proposed to impart anti-static effects to textiles of this type. However, none of these proposed agents is entirely satisfactory but all are deficient in effectiveness either initial or sustained and are also deficient with respect to resistance to washing and dry cleaning.

It is, therefore, an object of this invention to provide a method for the treatment of textile materials, particularly synthetic textile materials, whereby the textile materials are provided with a soft efficiently anti-static finish or dressing which has good resistance to washing or laundering or dry cleaning.

It is a further object of this invention to provide textile materials particularly synthetic textile materials which have a soft anti-static finish which is characterized by good resistance to washing, laundering or dry cleaning.

It is a further object of this invention to provide textile finishing agents which provide a durable anti-static finish on textile materials.

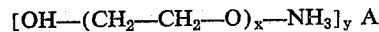
Other objects of this invention will appear from the following detailed description.

In accordance with the present invention it has been discovered that textile materials may be endowed with improved anti-static properties by treatment with an aqueous dispersion of a polyglycolamine salt, which salts will be further described hereinafter. (As used in this specification and appended claims, "dispersion," "dispersible" and related terms apply to true molecular distribution; i.e., solution as well as colloidal or near colloidal distribution.) When a textile material is treated with such a dispersion and dried, it was found to possess improved anti-static properties over those attainable using

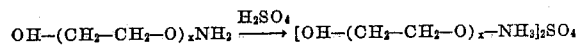
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well known commercially available so-called anti-static agents.

The polyglycolamine salts used in the method of this invention may be represented by the following general formula:



wherein x is an integer from 1 to 50, y is an integer from 1 to 3 and A is an anion of a strong mineral acid selected from the group consisting of I^- , Cl^- , Br^- , NO_3^- , $\text{SO}_4^{=}$ and $\text{PO}_4^{=}$. These polyglycolamine salts may be prepared in any convenient manner known to one skilled in the art. For example, a preferred polyglycolamine salt to be utilized in this invention is a polyglycolamine sulfate which may be prepared by sulfating a polyglycolamine thus:



The preparation of these type compounds is well known and does not form any part of the present invention.

These polyglycolamine salts may be applied to the textile materials in any convenient manner; however, it is preferred to apply the polyglycolamine salt from a single bath. This may be accomplished, for example, by treating the textile material with an aqueous dispersion of the polyglycolamine salt and thereafter drying the material. It has been found that an aqueous dispersion containing from about 0.2 percent to about 5.0 percent by weight of the polyglycolamine salt will cause a deposition of from about 0.12 percent by weight to about 5.8 percent by weight of the salt on the dried textile material. Obviously a more dilute or more concentrated aqueous dispersion could be utilized in this invention; however, from an economic and practical standpoint a 0.2 percent to 5.0 percent dispersion will consistently deposit a sufficient amount of the polyglycolamine salt on the textile material to render the same anti-static.

The term "textile materials" as employed in this specification and appended claims includes unspun fibers, both natural and synthetic staple fibers, yarns or continuous filaments and woven and knitted fabrics.

The term "textile materials" as employed in this specification and appended claims also includes natural and artificial textile fibers, yarns and fabrics comprising cotton, linen and other natural cellulose textile materials, viscose and cuprammonium and other regenerated cellulose textile material; wool casein, alpaca, and other natural or synthetic protein textile materials; cellulose acetate and other cellulose derivative textile materials; and nylon, vinyl chloridevinyl acetate copolymers, polyvinylidene polymers, acrylonitrile polymers and other synthetic polymer or condensation product textile materials.

The amount of polyglycolamine salt applied to the textile material may vary from 0.12 percent by weight to 5.8 percent by weight of the dried textile material; however, a deposition of from about 0.64 percent to 1.19 percent is most preferred in the practice of this invention. Within this range of deposition, an amount of polyglycolamine salt will be impregnated in the textile fabric to render the fabric both anti-static and resistant to washing, laundering and dry cleaning.

The above amounts of polyglycolamine salt are readily applied to the textile material by any of the procedures utilized in textile wet processing machinery. For example, the textile material may be immersed in, dipped in, sprayed with or roll coated with an aqueous dispersion containing from about 0.5 percent by weight to about 1.0 percent by weight of the polyglycolamine salt. The textile material is thereafter padded or centrifuged or the like to remove excess liquid and there is deposited from about 0.64 percent by weight to about 1.19 percent

by weight of the polyglycolamine salt on the textile material which is thereafter dried and tested for antistatic properties.

The temperature of the dispersion to be added to the textile may vary over a wide range; however, a temperature of from about 60° F. to about 120° F. has been found quite satisfactory in the application of this invention.

As stated above, the dried material is tested for antistatic properties in a specific test described below. In this test a resistance of 100 megohms was arbitrarily designated as the dividing point between a poor and a good anti-static agent. In the application of this test the resistance of a treated textile material to the passage of an electric current is measured. To facilitate this measurement a pair of electrodes are placed on the textile material, which electrodes are attached to a megohmmeter. When an electric current is passed through the megohmmeter the resistance is read directly from the meter. A more specific description of the apparatus used follows.

To a flat piece of Lucite 2" x 2" x 1/4" is attached two 1/8" x 1/8" x 1" copper strips which serve as electrodes. To the flat piece of Lucite is attached a Lucite rod 1/2" x 5" which serves as a handle. Attached to the electrodes (copper strips) and through the Lucite plate are insulated wire leads. These leads are carried through flexible metallic sheathing and terminate in jack-type connections which are inserted into a megohmmeter. The electrodes (copper strips) are so centered that they are parallel and exactly one inch apart. This described apparatus is placed perpendicular to a portion of the treated textile material and an electric current is allowed to pass through the system. The observance of the resistance is an indication of the anti-static properties of the treated textile material tested. It has been noted that the lower the resistance obtained the less tendency the treated textile material will possess to build up and maintain a static charge of electricity. In other words, a treated textile material indicating a low resistance (less than 100 megohms) in this test will be substantially anti-static.

The following examples are intended as illustrative of the present invention and are not to be construed limitative thereof.

EXAMPLE I

A polyglycolamine sulfate was prepared by reacting 25 g. of a polyglycolamine (mol. wt.=165) with 13.4 g. of 96.8 percent sulfuric acid. The resulting dispersion upon dilution with 25.0 cc. of water was found to have a pH of 7.5 and there was contained in the aqueous dispersion 52.9 percent by weight of the polyglycolamine sulfate. From this concentrated dispersion there was prepared five different more dilute dispersions containing 0.5 percent, 1.0 percent, 2.0 percent, 5.0 percent and 10.0 percent by weight of the polyglycolamine sulfate respectively.

Five 6" x 6" swatches, labeled consecutively 1 to 5, of cellulose acetate fabric were scoured with a commercial soap and water, rinsed and oven dried at 140° F. until dry to the touch. Each dispersion was then heated to 80° F. and the five swatches were treated with the dispersions of the polyglycolamine sulfate in the following manner: swatch number 1 was dipped into the dispersion containing 0.5 percent by weight of the polyglycolamine sulfate; swatch number 2 was dipped into the dispersion containing 1.0 percent by weight of the polyglycolamine sulfate, swatch number 3 was dipped into the dispersion containing 2.0 percent by weight of the polyglycolamine sulfate, swatch number 4 was dipped into the dispersion containing 5.0 percent by weight of the polyglycolamine sulfate and swatch number 5 was dipped into the dispersion containing 10.0 percent by weight of the polyglycolamine sulfate. The five swatches so treated were passed through squeeze rolls to remove

any excess liquid and oven dried at 140° F. The dried swatches were then transferred to a desiccator and placed in an area of constant humidity (50 percent \pm 1 percent) where they remained for at least 24 hours and finally the resistance of the material was measured using the above described apparatus. The results obtained are presented in Table I.

Table I

Swatch Number	Concentration of Dispersion	Percent Salt Deposited on Fabric	Resistance in Megohms
1	0.5% by wt. polyglycolamine sulfate.	0.64	30.0
6	0.5% by wt. monoethanolamine sulfate.	0.56	>400
11	0.5% by wt. triethanolamine sulfate.	0.06	390
16	0.5% by wt. tetraethanolamine sulfate.	unmeasurable.	>400
2	1.0% by wt. polyglycolamine sulfate.	1.19	17.0
7	1.0% by wt. monoethanolamine sulfate.	1.06	40.0
12	1.0% by wt. triethanolamine sulfate.	0.44	105
17	1.0% by wt. tetraethanolamine sulfate.	unmeasurable.	>400
3	2.0% by wt. polyglycolamine sulfate.	2.37	8.8
8	2.0% by wt. monoethanolamine sulfate.	2.12	19.0
13	2.0% by wt. triethanolamine sulfate.	1.25	35.0
18	2.0% by wt. tetraethanolamine sulfate.	0.30	>400
4	5.0% by wt. polyglycolamine sulfate.	5.81	3.5
9	5.0% by wt. monoethanolamine sulfate.	3.10	4.0
14	5.0% by wt. triethanolamine sulfate.	3.50	7.1
19	5.0% by wt. tetraethanolamine sulfate.	0.50	300
5	10.0% by wt. polyglycolamine sulfate.	-----	2.2
10	10.0% by wt. monoethanolamine sulfate.	-----	1.1
15	10.0% by wt. triethanolamine sulfate.	-----	2.0
20	10.0% by wt. tetraethanolamine sulfate.	-----	30.0

EXAMPLE II

There was prepared five separate dispersions of monoethanolamine sulfate containing 0.5 percent, 1.0 percent, 2.0 percent, 5.0 percent and 10.0 percent by weight respectively. Another five 6" x 6" swatches labeled consecutively 6 to 10, of cellulose acetate fabric were scoured with a commercial soap and water, rinsed and oven dried at 140° F. until dry to the touch.

Each dispersion was then heated to 80° F. and the five swatches were treated with the dispersions of the monoethanolamine sulfate in the following manner: swatch number 6 was dipped into the dispersion containing 0.5 percent by weight of the monoethanolamine sulfate, swatch number 7 was dipped into the dispersion containing 1.0 percent by weight of the monoethanolamine sulfate, swatch number 8 was dipped into the dispersion containing 2.0 percent by weight of the monoethanolamine sulfate, swatch number 9 was dipped into the dispersion containing 5.0 percent by weight of the monoethanolamine sulfate and swatch number 10 was dipped into the dispersion containing 10.0 percent by weight of the monoethanolamine sulfate. The five swatches so treated were passed through squeeze rolls to remove any excess liquid and oven dried at 140° F. The dried swatches were then transferred to a desiccator and placed in an area of constant humidity (50 percent \pm 1 percent) where they remained for at least 24 hours and finally the resistance of the fabric was measured using the above described apparatus. The results obtained are presented in Table I.

EXAMPLE III

There was prepared five separate dispersions of triethanolamine sulfate containing 0.5 percent, 1.0 percent,

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2.0 percent, 5.0 percent and 10.0 percent by weight respectively. Another five 6" x 6" swatches, labeled consecutively 11 to 15, of cellulose acetate fabric were scoured with a commercial soap and water, rinsed and oven dried at 140° F. until dry to the touch.

Each dispersion was then heated to 80° F. and the five swatches were treated with the dispersions of the triethanolamine sulfate in the following manner: swatch number 11 was dipped into the dispersion containing 0.5 percent by weight of the triethanolamine sulfate, swatch number 12 was dipped into the dispersion containing 1.0 percent by weight of the triethanolamine sulfate, swatch number 13 was dipped into the dispersion containing 2.0 percent by weight of the triethanolamine sulfate, swatch number 14 was dipped into the dispersion containing 5.0 percent by weight of the triethanolamine sulfate and swatch number 15 was dipped into the dispersion containing 10.0 percent by weight of the triethanolamine sulfate. The five swatches so treated were passed through squeeze rolls to remove any excess liquid and oven dried at 140° F. The dried swatches were then transferred to a desiccator and placed in an area of constant humidity (50 percent ± 1 percent) where they remained for at least 24 hours and finally the resistance of the fabric was measured using the above described apparatus. The results obtained are presented in Table I.

EXAMPLE IV

There were prepared five separate dispersions of tetraethanolamine sulfate containing 0.5 percent, 1.0 percent, 2.0 percent, 5.0 percent, and 10.0 percent by weight respectively. Another five 6" x 6" swatches, labeled consecutively 16 to 20, of cellulose acetate fabric were scoured with a commercial soap and water, rinsed and oven dried at 140° F. until dry to the touch.

Each dispersion was then heated to 80° F. and the five swatches were treated with the dispersions of the tetraethanolamine sulfate in the following manner: swatch number 16 was dipped into the dispersion containing 0.5 percent by weight of the tetraethanolamine sulfate, swatch number 17 was dipped into the dispersion containing 1.0 percent by weight of the tetraethanolamine sulfate, swatch number 18 was dipped into the dispersion containing 2.0 percent by weight of the tetraethanolamine sulfate, swatch number 19 was dipped into the dispersion containing 5.0 percent by weight of the tetraethanolamine sulfate and swatch number 20 was dipped into the dispersion containing 10.0 percent by weight of the tetraethanolamine sulfate. The five swatches so treated were passed through squeeze rolls to remove any excess liquid and oven dried at 140° F. The dried swatches were then transferred to a desiccator and placed in an area of constant humidity (50 percent ± 1 percent) where they remained for at least 24 hours and finally the resistance of the fabric was measured using the above described apparatus. The results obtained are presented in Table I.

Table I clearly shows the desirable anti-static properties of a synthetic textile material treated according to this invention. The comparison data presented in Table I shows that a synthetic textile material treated with a 0.5 percent to 1.0 percent dispersion of a polyglycolamine salt and having deposited thereon about 0.64 percent to 1.19 percent of a polyglycolamine salt will possess far superior anti-static properties over those obtained using previously disclosed so-called anti-static agents. With the exception of a cellulose diacetate swatch treated with a 1.0 percent by weight dispersion of a monethanolamine sulfate (swatch number 7) all other compounds tested within the preferred concentration range of this invention failed to indicate a satisfactory treatment of the synthetic textile material to render it anti-static within the definition of this invention. Obviously as the concentration of the dispersions of the various salts used in obtaining the experimental data presented in Table I

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was increased the anti-static properties of the mono-, tri- and tetraethanol sulfates increased until at a concentration of a dispersion of a salt of 10.0 percent by weight all salts including the compounds utilized in this invention rendered the fabrics so treated efficiently anti-static. However in the preferred concentration ranges of this invention the superiority of the polyglycolamine salts as compared to conventional anti-static agents is clearly demonstrated.

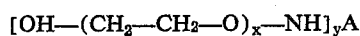
Textile materials prepared from cellulose diacetate have been utilized to demonstrate the superior anti-static effect of a polyglycolamine salt since cellulose diacetate fabrics are highly susceptible to the accumulation of static electrical charges. It is to be understood, however, that all natural and synthetic fibers described hereinabove are applicable to be treated according to this invention.

Although the usefulness of this invention lies in providing natural and synthetic textile fibers with desired anti-static properties, it also may be pointed out that the polyglycolamine salts of this invention also impart an improved "hand" to the textile material.

Various changes and modifications in the processes and textile materials herein described may be made as will be apparent to those skilled in the art to which this invention appertains without departing from the spirit and intent of this invention. It therefore is to be understood that the present invention is not to be limited except by the scope of the appended claims.

I claim:

1. A method of treating textile materials to render the same anti-static which comprises treating said textile materials with a dilute aqueous dispersion of a salt of the general formula:



wherein x is an integer from 1 to 50, y is an integer from 1 to 3 and A is an anion of a strong mineral acid, whereby a portion of said salt is deposited on said textile materials and thereafter drying said textile materials.

2. A method of treating synthetic textile materials to render the same anti-static which comprises treating said textile materials with a dilute aqueous dispersion containing from about 0.2 percent to 5.0 percent by weight of a salt of the general formula:



wherein x is an integer of from 1 to 50, y is an integer from 1 to 3 and A is an anion of a strong mineral acid whereby a portion of said salt is deposited on said textile materials and thereafter drying said textile materials.

3. A method of treating synthetic textile materials to render the same anti-static which comprises treating said textile materials with a dilute aqueous dispersion containing from about 0.5 percent to 1.0 percent by weight of a salt of the general formula:



wherein x is an integer of from 1 to 50, y is an integer from 1 to 3 and A is an anion of a strong mineral acid whereby a portion of said salt is deposited on said textile materials and thereafter drying said textile materials.

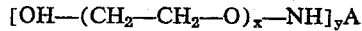
4. A method of treating synthetic textile materials to render the same anti-static which comprises treating said textile materials with a dilute aqueous dispersion containing from about 0.5 percent to 1.0 percent of a salt of the general formula:



wherein x is an integer from 1 to 50, y is an integer from 1 to 3 and A is an anion of a strong mineral acid selected from the group consisting of I^- , Cl^- , Br^- , NO_3^- , SO_4^{2-} , and PO_4^{3-} whereby a portion of said salt is deposited on said textile fabric ranging in amount between 0.64 percent and 1.19 percent by weight of said textile materials and thereafter drying said textile materials.

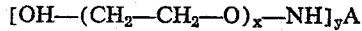
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5. Textile materials comprising a major proportion of synthetic textile materials and impregnated with a salt of the general formula:



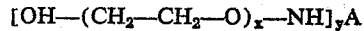
wherein x is an integer of from 1 to 50, y is an integer of from 1 to 3 and A is an anion of a strong mineral acid, in an amount ranging between 0.12 percent and 5.8 percent by weight of said textile materials.

6. Textile materials comprising a major proportion of synthetic textile materials and impregnated with a salt of the general formula:



wherein x is an integer of from 1 to 50, y is an integer of from 1 to 3 and A is an anion of a strong mineral acid, in an amount ranging between 0.64 percent and 1.19 percent, by weight of said textile materials.

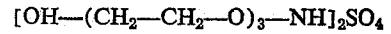
7. Textile materials comprising a major proportion of synthetic textile materials and impregnated with a salt of the general formula:



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wherein x is an integer of from 1 to 50, y is an integer from 1 to 3 and A is an anion of a strong mineral acid selected from the group consisting of I^- , Cl^- , Br^- , NO_3^- , $\text{SO}_4^{=}$, and $\text{PO}_4^{=}$ in an amount ranging between 0.64 percent and 1.19 percent by weight of said textile material.

8. Textile materials comprising a major proportion of synthetic textile materials and impregnated with a polyglycolamine sulfate having the following formula:



in an amount ranging between 0.64 percent and 1.19 percent by weight of said textile materials.

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