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POLYAMIDE FIBERS TREATED WITH AN AMPHOTERIC ACTIVATOR

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This is a continuation-in-part of application Serial No. 223,512, filed September 13, 1962, now abandoned, in the name of Julian J. Hirshfeld for "Activating Agents."

This invention is concerned with a finish for synthetic fiber flock. More specifically, this invention is concerned with a finish for synthetic linear polyamide fibers which will render the flock of these fibers active in an electrostatic field and also prevent the flock from adhering together in clumps, thus providing for a free flow of the flock in the electrostatic field.

Of major importance today in the field of textiles is the electrostatic method of flocking. Two electrodes are used with a high potential difference between them and when the flock is introduced into this charged atmosphere, it is attracted to one of the electrodes (it is charged by one of the electrodes and attracted toward the other, oppositely charged electrode). The base obviously is coated with an adhesive and this is positioned in front of one of the electrodes to catch the flock as it is attracted to the electrode. These fibers or flock automatically line themselves up along the lines of force in the electrostatic field and the base is positioned at right angles to this direction. Thus with the use of the electrostatic method, the flock must be capable of taking an electrostatic charge; otherwise, the electrostatic field will not activate the flock in any direction.

In the past, using methods of the prior art, it has thus been difficult to secure an even deposit of the flock upon the base. The flock has a tendency to gather into tight bundles or lumps and thus be deposited in clumps not evenly spaced, thus giving an appearance of a lot of flock in one area and very little in another area of the base. This is objectionable as it is not commercially acceptable to use an uneven appearing flock.

In order to render the flock effective, it is necessary that it be treated with a special finish activator. The flocking of natural fibers, such as cotton and wool, do not present a difficult problem because electrolytes, for example salt, applied in aqueous solution would activate these fibers in a satisfactory manner. This appears to be due to their inherent hydrophilic nature, and when thus treated, transforms the flock into good conductors. However, the above is not correct when concerned with synthetic fiber flock which is of hydrophobic nature. In addition, synthetic fiber flock has a tendency to clog, forming lumps which hinder an effective even flocking operation. In order to achieve the best results, it is necessary that the flock be capable of taking and holding a high electrostatic charge, yet remain loose, dry and free flowing.

An object of this invention is to provide a finish which will render synthetic fiber flock active in an electrostatic field.

Another object of this invention is to provide a finish for the flock of polyamide fibers which will render the fibers active in an electrostatic field.

Another object of this invention is to provide a finish which will render the flock of synthetic fibers highly chargeable in an electrostatic field.

Another object of this invention is to provide a finish for the flock of synthetic polyamide fibers which will enable the flock to deposit in a very even distribution on the base in an electrostatic field.

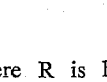
Other objects and advantages of this invention will be readily apparent from the detailed description which follows hereafter.

Generally the objects of this invention are accomplished by treating the flock of polyamide fibers with an amphoteric compound containing both a sodium sulfonate group as well as a tertiary amino group or a mixture of said amphoteric compound and sodium chloride.

More specifically, the polyamide flock is treated for 5 minutes to 60 minutes, with the preferred being 20 minutes, at a temperature of from 70° to 200° F., with the preferred being 130° F., with varying amounts, based upon the weight of the flock, of an amphoteric compound, containing both a sodium sulfonate group as well as a tertiary amino group or a mixture of said amphoteric compound and sodium chloride. The particular amphoteric compound may be an aqueous dispersion of a gamma sodium sulfonate of an ethoxylated stearamine, the specific compound being the sodium salt of N,N'-diethoxylamino-1-octadecane-3-sulfonic acid. The flock, once it has been treated with the above identified aqueous solutions, is then dried and maintained under conditions of 30 percent to 80 percent of relative humidity with the preferred being 75 percent relative humidity at room temperature. Not only may the compound identified above be used as an activating agent for the polyamide flock, but the compound along with common table salt in a mixture may be used to activate the polyamide flock.

The amount of common sodium chloride used may vary from a 5 percent solution to a completely saturated solution (35.7 percent). In addition, varying amounts as pointed out above of the amphoteric compound may be used varying from 1.0 percent to 100 percent, with the preferred being 25 percent, all based upon the weight of the flock. In addition, varying electrostatic field intensities may be used such as 10,000 volts to 100,000 volts. In reality, there is really no minimum amount of intensity or a maximum amount of intensity, this being determined by the type of flocking desired and also the natural physical or electrical limitations of equipment used in creating the field of intensity.

While this invention has been generally directed to synthetic fiber flock, it is especially useful in activating flock of fibers composed of polyamide polymers. Simple linear polyamides which are normally insoluble in alcohol but are soluble in phenols 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, 2,071,255 and 2,130,948. The term polycarbonamide means polymers having recurring units of formula:



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, tertamethylenediamine suberic acid, tetramethylenediamine with sebacic acid, hexamethylene diamine 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 following examples are intended to illustrate this invention more fully and are not intended to limit the scope of the invention, for it is possible to effect many modifications therein. In the examples, all parts and percents are by weight unless otherwise indicated.

Example 1

A sample of 10 grams of polyhexamethylene diamine adipamide fibers were scoured under normal conventional conditions and dried. The fibers were cut into four millimeter lengths. This flock was then placed in a nylon knitting bag and immersed in an aqueous bath, ratio of 25 to 1, containing 20 percent of an amphoteric compound containing both a sodium sulfonate group and a tertiary amino group, specifically a 33 percent aqueous dispersion of the sodium salt of N,N-diethoxylamino-1-octadecane-3-sulfonic acid, based on the weight of the flock. Bath solids content was 6.7 percent. The flock remained in the bath for 20 minutes at 130° F. The thus treated flock was then removed from the bath, lightly centrifuged, dried at 220° F. and conditioned overnight in a relative humidity atmosphere of 75 percent under room temperature which approximated 75° F. Total activator solids on weight of fiber was 0.45 percent. A sample of the above conditioned flock in the amount of 0.5 gram was then placed upon a plate in a Portable Industry Laboratory Flocking Unit, Model A (Dekor Flocking Company). The distance between the two plates of this flocking machine was four inches. An electrical potential of 20,000 volts was set up and this flock cleared the lower plate within an average of 20 seconds. There was no crystal formation on the flock, its activity was good and the flow was good.

Flow is a subjective type test in which it is determined whether the flock will lump together when it is placed in motion or whether the individual flock will be separated and move evenly and smoothly through any space.

Example 2

The exact procedure in Example 1 was followed except the flock remained in the treating bath for 15 minutes at 200° F. Total activator solids on weight of fiber was 0.52 percent. The flock cleared the lower plate of the flocking machine within an average of 6 seconds and the activity was good. There was no crystal formation on the flock and the flow of the flock was good.

Example 3

The exact procedure of Example 1 was followed except the treating bath was composed of 30 percent of the amphoteric compound identified hereinbefore. Bath solids was 10 percent and activator solids on weight of fiber was 0.70 percent. The flock cleared the lower plate of the flocking machine within an average of 15 seconds and the activity was good. There was no crystal formation on the flock and the flow of the flock was good.

Example 4

The exact procedure of Example 3 was followed except the flock was maintained in the treating bath for 15 minutes at 200° F. Activator solids on weight of fiber was 0.75 percent. The flock cleared the lower plate of the flocking machine with an average of 8 seconds and the activity was good. There was no crystal formation on the flock and the flow of the flock was good.

Example 5

The exact procedure of Example 1 was followed except the treating bath was composed of 30 percent of the amphoteric compound (net solids 10 percent) and 25 percent of common salt (sodium chloride). Activator solids on weight of fiber was 1.6 percent. The flock cleared the lower plate of the flocking machine within an average of 6 seconds and the activity was good. There was no crystal formation on the flock and the flow of the flock was good.

Example 6

The exact procedure of Example 1 was followed except the treating bath was composed of 30 percent of the amphoteric compound (net solids 10 percent) and 5 percent common salt (sodium chloride) and the flock was maintained in the treating bath for 15 minutes at 200° F. Activator solids on weight of fiber was 0.90 percent. The flock cleared the lower plate of the flocking machine within an average of 5 seconds and the activity was good. There was no crystal formation on the flock and the flow of the flock was good.

Example 7

The exact procedure of Example 1 was followed except the treating bath was composed of 20 percent of the amphoteric compound (net solids 6.7 percent) and 10 percent common salt (sodium chloride). Activator solids on weight of fiber was 1.0 percent. The flock cleared the lower plate of the flocking machine in an average of 5 seconds and the activity was good. There was no crystal formation on the flock and the flow of the flock was good.

Example 8

The exact procedure of Example 1 was followed except the treating bath was composed of 15 percent of the amphoteric compound, 5 percent net solids, and brine, wherein the water of the bath was approximately saturated with sodium chloride (around 35 percent). Activator solids on weight of fiber was 1.6 percent. The flock cleared the lower plate of the flocking machine within an average of 3 seconds and the activity was fairly good. There was no crystal formation on the flock and the flow of the flock was good.

Example 9

The exact procedure of Example 1 was followed except the treating bath was composed of 10 percent of the amphoteric compound, 3.3 percent net solids, and brine, and the flock was maintained in the treating bath for 15 minutes at 200° F. Activator solids on weight of fiber was 1.7 percent. The flock cleared the lower plate of the flocking machine with an average of 2 seconds and the flow of the flock was good.

Example 10

The exact procedure of Example 1 was followed except the treating bath was composed of 20 percent of the amphoteric compound, 6.7 percent net solids, and brine. Activator solids on weight of fiber was 1.7 percent. The flock cleared the lower plate of the flocking machine within an average of 2 seconds and the activity was good. There was no crystal formation on the flock and the flow of the flock was good.

Example 11

The exact procedure of Example 1 was followed except the treating bath was composed of 30 percent of the amphoteric compound, 10 percent net solids and brine and the flock was maintained in the treating bath for 15

minutes at 200° F. Activator solids on weight of fiber was 1.7 percent. The flock cleared the lower plate of the flocking machine within an average of 2 seconds and the activity was good. There was no crystal formation on the flock and the flow of the flock was good.

It will be noted that the amount of the activating agent added to the flock is, by weight, in the range of about 0–2 percent. The amount of solids in the bath, by weight, varies up to about 10 percent.

While the above examples were flocked in lengths of four millimeters from an 18 denier fiber, as of the present time, flock varying from tiny particles up to 12 millimeters have been successfully processed through this flocking operation. In addition, while the denier of the flock in the above examples is 18, there is reality, no limitation of the size of denier which may be used. With flock cut from a fiber of a denier smaller or larger than 18, it would only be necessary to have some adjustments in the flocking machine which a person skilled in the flocking art would easily accomplish.

The above examples were run under normal textile control conditions which generally are around 50 to 75 percent relative humidity and from 70° F to 75° F.; however, the flocking operation will perform under conditions which are not within this preferred textile testing range. Further, the distance between the two plates in the flocking machine used in the above examples was four inches. Generally flocking machines operate with an electrical potential difference of from 10,000 to 100,000 volts.

Thus it is seen with the activating agents or finishes of the above examples of this invention, flock cut from polyamide fibers have been successfully activated so that they perform extremely well in the flocking process. By the term "activated," we means that the flock is rendered capable of taking and holding a high electrostatic charge. The time element in which the flock clears the plate has been very low and the flow of the flock with the above applied finishes has been very acceptable.

This invention has provided a finish for the flock of polyamide fibers which enable the polyamide fibers to enter successfully and extensively into the commercial field of flocking, which up to now has been generally dominated by the viscose rayon fibers along with natural occurring fiber, such as cotton, wool, etc.

It will be understood that changes and variations may be made in the present invention by one skilled in the art without departing with the spirit and scope thereof as defined in the appended claims.

What is claimed is:

1. A composition of matter composed of synthetic linear polyhexamethylene adipamide fibers and an activator consisting of a sodium salt of N,N-diethoxylamino-1-ocadecane-3-sulfonic acid, the amount of said sodium salt being up to 2 percent by weight of the fiber.

2. The composition of matter of claim **1** wherein the activator consists of a mixture of N,N-diethoxylamino-1-octadecane-3-sulfonic acid and sodium chloride, the amount of said activator being up to 2 percent by weight of the fiber.

References Cited

UNITED STATES PATENTS

2,086,544	7/1937	Dreyfus	117—114 X
2,153,358	4/1939	Whitehead	117—4 X
2,668,785	2/1954	Jefferson et al.	117—4 X
2,717,877	9/1955	Vitalis	252—8.7
2,798,044	7/1957	Vitalis	252—8.7
2,805,959	9/1957	Ewing	117—100 X
2,974,066	3/1961	Marcura et al.	117—138.8 X
2,992,126	7/1961	Roberts et al.	117—27 X
2,993,811	7/1961	Smith et al.	117—138.8 X

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