United States Patent Office

3,033,697

Patented May 8, 1962

3,033,697

MANUFACTURE OF PIGMENTED VISCOSE RAYON

Brewster B. Eskridge, Candler, N.C., assignor to American Enka Corporation, Enka, N.C., a corporation of Delaware

No Drawing. Filed May 31, 1960, Ser. No. 32,593

9 Claims. (Cl. 106—166)

This invention relates generally to the manufacture of pigmented viscose rayon articles such as filaments, threads, fibers, and the like and more particularly to the manufacture of solution-colored and/or delustered viscose rayon articles.

In the prior art, various methods have been proposed for the production of pigmented viscose rayon. If a solution-colored yarn is desired, it has been customary to incorporate one or more organic coloring pigments into the viscose spinning solution. One difficulty in such a method was that the fabrics obtained from such yarn exhibited dichroism. It was proposed in U.S. Patent No. 2,934,449 that the above could be overcome by the incorporation into the viscose spinning solution of a small amount of a substantially white inorganic delustering pigment.

If a delustered yarn is desired, it has been proposed that a relatively large amount, for example 1.0% to 5.0%, based on the weight of the cellulose in the viscose, be incorporated into the viscose spinning solution. Thus it was possible to obtain a dull luster yarn or a delustered and solution-colored yarn which depends upon the absence or presence of organic coloring pigments in the viscose solution.

Many difficulties have arisen in the use of the finely divided delustering pigments either alone or in conjunction with organic coloring pigments. They exhibit a strong tendency to settle out of the viscose solution. Also, they may agglomerate and clog the spinning orifices or cause weak spots in the yarn, thereby reducing the quality of the yarn. It has been proposed that dispersing agents be used to overcome these problems. However, difficulties have arisen here because they may be precipitated out of the viscose spinning solution by the metal cations present therein, or they may not be compatible with the organic coloring pigments or their dispersing agents. They may also be precipitated by the organic coloring pigments or their dispersing agents.

It is therefore an object of the present invention to provide a process for the production of pigmented viscose rayon that is free of the difficulties of the prior art.

It is a further object of the present invention to provide a viscose spinning solution for the manufacture of solution-colored viscose rayon.

A further object of this invention is to provide a viscose spinning solution for the manufacture of solution-colored rayon wherein settling out of the pigments is substantially eliminated.

Another object of the present invention is to provide a viscose spinning solution for the manufacture of solution-colored rayon wherein precipitation of the dispersing agent used therein does not occur.

An additional object of this invention is to provide a viscose spinning solution for the manufacture of delustered and/or solution-colored rayon wherein the pigments retain their finely dispersed state.

These and other objects of this invention are accomplished by providing a viscose spinning solution having dispersed therein an organic coloring pigment and/or a substantially white inorganic delustering pigment, a primary aliphatic amine containing 12 to 18 carbon atoms, and sodium N-coco beta-aminopropionate or disodium N-tallow beta-iminodipropionate. These propionates

are known in the trade as Deriphats and are N-substituted amino acid derivatives (beta-alanines). They are derived from the condensation of fatty primary amines and acrylic monomers. Coco denotes a mixture of fatty acids obtained from coconut oil, and tallow indicates a mixture of fatty acids obtained from tallow fat. These mixed fatty acids have straight carbon radicals ranging from about 8 to 24 carbon atoms with the individual amines in a distribution corresponding to the distribution of the fatty acids of the particular fat or oil.

By the introduction of the amine and propionate into the viscose spinning solution by way of the pigment dispersion, it was unexpectedly found that it was possible to produce a delustered and/or solution-colored viscose rayon yarn in which the pigments were finely dispersed. In addition, there was no evidence of a precipitate which could be attributed to the pigments or compounds. Also, there was a substantial reduction in the settling out of the delustering pigment.

The substantially white inorganic pigments that are contemplated by this invention are those commonly known as delustrants. Examples of these are titanium dioxide, tin oxide, barium sulfate, lead sulfate, and zinc oxide. The preferred compound is titanium dioxide. If dichroism is to be prevented in a lustrous solution-colored yarn, then it is only necessary that about 0.01–0.10%, based on the weight of the cellulose in the viscose (hereinafter referred as CIV) be dispersed in the viscose solution. However, if a delustered yarn is desired, then about 1.0 to 5.0% CIV should be used, and if a delustered and solution-colored yarn is desired, then normally about 0.1 to 1.0% CIV will be used. It should be understood that this invention is not restricted to any precise range since it is obvious that the proportions of the delustrant may vary within wide limits, depending on the pigment and the type of coloring material used.

The coloring materials contemplated by this invention are conventional organic coloring pigments. Examples of these are Microsol Yellow, Imperse Red, Stabaloid Black, Monolite Yellow. Monolite Red, Aquablack X, and Monastral Green. The amount of the coloring pigment will vary depending upon the desired color. The selection of the pigments and their amounts are easily determined by means known to those skilled in the art.

The aliphatic amines required for successful operation of this invention are those that contain 12 to 18 carbon atoms, as for example, lauryl amine and oleyl amine. It is only necessary that very small amounts of the amine be incorporated into the viscose spinning solution. For instance, if 0.01 to 0.1% (CIV) titanium dioxide is used, normally 0.0005 to 0.005% (CIV) will be sufficient. However, if 1.0 to 5.0% (CIV) of titanium dioxide is to be used, then normally 0.05 to 0.25% (CIV) will be sufficient to aid in the prevention of settling out of the pigments and precipitation of any of the salts present therein. It should be understood that larger or smaller amounts may be used without departing from this invention.

The second organic compound contemplated by this invention for use in conjunction with the above pigments is sodium N-coco beta-aminopropionate or disodium N-tallow beta-iminodipropionate. It is only necessary that very small amounts of these compounds be used. For instance, if 0.01 to 0.1% (CIV) titanium dioxide is used, normally 0.0006 to 0.006% (CIV) will be sufficient. However, if 1.0 to 5.0% (CIV) titanium dioxide is to be used, then normally 0.06 to .30% (CIV) will be sufficient to aid in the prevention of settling out of the pigments and precipitation of any of the salts present therein. It should be understood that larger or smaller amounts may be used without appreciably affecting the results.

The dispersion of these pigments and organic compounds in the viscose solution may be accomplished in any conventional manner. For instance, a stable slurry of the inorganic pigment and two organic compounds is prepared by uniformly dispersing the same in a liquid, for example, water. The pigment slurry may be prepared by grinding the inorganic pigment and two organic compounds in the presence of water in a pebble mill, a ball mill, a colloid mill such as a Charlotte mill, or a homo mixer. The resulting dispersion does not exhibit any settling out for several days.

If a delustered viscose rayon yarn is desired, an appropriate aliquot may be withdrawn from this slurry and incorporated into the viscose spinning solution. Normally the same will be injected into a flowing stream of viscose and thereafter the viscose and pigment dispersion are passed through a conventional mixing stage wherein the same are thoroughly mixed throughout the viscose spinning solution. Thereafter the pigmented viscose solution is extruded into a conventional coagulating bath to form filaments therefrom.

An organic coloring pigment may be separately incorporated into the viscose solution before or after the above pigment dispersion. It may also be incorporated into the pigment dispersion and thereafter injected or otherwise incorporated into the viscose spinning solution. The amount of organic pigments used will, of course depend upon the color and shade desired. In addition, the amount of inorganic pigment used will depend upon whether only dichroism is to be prevented or that a delustered and solution-colored yarn is to be obtained.

It should be understood that any conventional make-up of the pigments and organic compounds may be used. In addition, any method of dispersing the same in the viscose spinning solution may be used without departing from this invention.

The following examples are included merely for the purpose of illustration and are not to be considered as limiting the scope of this invention.

Example I

An aqueous dispersion of 20% titanium dioxide, 1.0% primary lauryl amine, and 1.25% disodium N-tallow beta-iminodipropionate was prepared by mixing the same in a homo mixer. A predetermined portion of the dispersion was withdrawn and was added to a viscose solution prepared in the usual manner. The formulation was 1.0% (CIV) titanium dioxide, 0.05% (CIV) primary lauryl amine, and 0.06% (CIV) disodium N-tallow beta-iminodipropionate. The delustered viscose was extruded through a spinneret into an acid coagulating bath. The finished filaments were closely wound on a viewing card and exhibited a dull luster.

The pigment dispersion did not settle on standing, and the titanium dioxide was thoroughly dispersed in the resulting yarn. The spinnability of this viscose was normal, and the quality was normal.

Example II

An aqueous dispersion of titanium dioxide was prepared as in Example I. A predetermined portion of this dispersion was withdrawn and was added to a coloring pigment suspension. After thorough mixing, a predetermined amount of the resulting dispersion was added to a viscose solution prepared in the usual manner. The formulation was 0.7% (CIV) Monolite Yellow, 0.725% (CIV) Monolite Red, 0.35% (CIV) Aquablack X, 0.45% (CIV) titanium dioxide, 0.0225% (CIV) primary lauryl amine, and 0.027% (CIV) disodium N-tallow beta-iminodipropionate. The solution-colored viscose was extruded through a spinneret into an acid coagulating bath. The finished filaments so produced were closely wound on a viewing card. When the filaments were viewed at different angles, there was no detectably different surface color perceptions and the color was autumn brown.

The pigment dispersion did not settle on standing and there was no precipitate formed. In addition, the yarn was uniformly colored and the pigments were thoroughly dispersed throughout the yarn. The spinnability of the viscose and the quality of the yarn were normal.

Example III

An aqueous dispersion of 20% titanium dioxide, 1.0% primary lauryl amine, and 1.25% disodium N-tallow beta-iminodipropionate was prepared by mixing the same in a homo mixer. The dispersion was allowed to stand for seven days, and there was no settling of the titanium dioxide which remained thoroughly dispersed therein.

A portion of this dispersion was added to several standard organic coloring suspensions and there was no settling or precipitation evident.

Example IV

An aqueous dispersion was prepared as in Example III except sodium N-coco beta-aminopropionate was substituted for the disodium compound. This dispersion was allowed to stand for seven days and the result was the same as in Example III. Also there was no settling or precipitation when a portion of this dispersion was added to standard organic coloring suspensions.

Example V

An aqueous dispersion was prepared in the same manner as in Example III, except primary oleyl amine was substituted for the lauryl amine. This dispersion did not settle after seven days nor did it exhibit any precipitation when mixed with standard organic coloring suspensions.

It can be seen from the foregoing examples that delustering pigment dispersions and/or solution-coloring dispersions may be prepared, which dispersions do not settle on long standing, nor is any precipitate formed. This permits the yarn producer to prepare large amounts of the pigment dispersions which may be stored for several days. This results in substantial economy in his operations. In addition, the same do not lower the spinnability of the viscose or the quality of the yarn, and at the same time produce a uniformly delustered and/or solution-colored viscose rayon yarn.

It should be understood that various modifications may be made in this invention without departing from its spirit and scope, which is to be limited only by the following claims.

What is claimed is:

1. A viscose spinning solution for the manufacture of rayon filaments having dispersed therein 0.01 to 5.0% (CIV) of a finely divided substantially white inorganic delustering pigment, 0.0005 to 0.25% (CIV) of a primary aliphatic amine containing 12 to 18 carbon atoms, and 0.0006 to 0.3% (CIV) of a compound selected from the group consisting of disodium N-tallow beta-iminodipropionate and sodium N-coco beta-aminopropionate.

2. A viscose spinning solution according to claim 1 wherein said inorganic pigment is titanium dioxide.

3. A viscose spinning solution according to claim 1 wherein said compound is disodium N-tallow beta-iminodipropionate.

4. A viscose spinning solution according to claim 1 wherein said compound is sodium N-coco beta-aminopropionate.

5. A viscose spinning solution according to claim 1 wherein the primary aliphatic amine is lauryl amine.

6. A viscose spinning solution according to claim 1 wherein the primary aliphatic amine is oleyl amine.

7. A viscose spinning solution for the manufacture of rayon filaments having dispersed therein an organic coloring pigment, 0.01 to 5.0% (CIV) of a finely divided substantially white inorganic delustering pigment, 0.0005 to 0.25% (CIV) of a primary aliphatic amine containing 12 to 18 carbon atoms, and 0.0006 to 0.3% (CIV) of a

compound selected from the group consisting of disodium N-tallow beta-iminodipropionate and sodium N-coco beta-aminopropionate.

8. A viscose spinning solution for the manufacture of delustered rayon filaments having dispersed therein 1.0 to 5.0% (CIV) titanium dioxide, 0.05 to 0.25% (CIV) of a primary aliphatic amine containing 12 to 18 carbon atoms, and 0.06 to 0.3% (CIV) of a compound selected from the group consisting of disodium N-tallow beta-iminodipropionate and sodium N-coco beta-aminopropionate.

9. A viscose spinning solution for the manufacture of solution-colored rayon filaments having dispersed therein an organic coloring pigment, 0.01 to 1.0% (CIV) titanium

dioxide, 0.0005 to 0.05% (CIV) of a primary aliphatic amine containing 12 to 18 carbon atoms, and 0.0006 to 0.06% (CIV) of a compound selected from the group consisting of disodium N-tallow beta-iminodipropionate and sodium N-coco beta-aminopropionate.

References Cited in the file of this patent

UNITED STATES PATENTS

2,481,692	Schlosser et al.	Sept. 13, 1949
2,867,540	Harris	Jan. 6, 1959
2,927,091	Liggett	Mar. 1, 1960
2,934,449	Jones	Apr. 26, 1960