Patented Mar. 13, 1934 1,951,094

UNITED STATES PATENT OFFICE

1,951,094

METHOD OF MAKING ARTIFICIAL SILK AND PRODUCT THEREOF

Theodoor Koch, Arnhem, Netherlands, assignor, by mesne assignments, to American Enka Corporation, Wilmington, Del., a corporation of Delaware

No Drawing. Application August 3, 1931, Serial 554,950. In Germany February 21, 1931

12 Claims. (Cl. 18—54)

The invention relates to artificial silk having matt appearance. An object of the invention is an advantageous process which will produce a very desirable artificial silk of that description and the invention consists in the process and the product described by or included within the terms or scope of the appended claims.

Describing the invention particularly in connection with the manufacture of viscose silk, it is characterized by the incorporation in the artificial silk thread of an inorganic inert substance such for example as barium sulfate, strontium sulfate and calcium sulfate and in particular, the distribution thereof in cell-like spaces and not as has been the case in prior attempts, the distribution of fine particles uniformly in the thread. Heretofore, it has been proposed to dissolve soluble barium compounds in the viscose solution and use the acid precipitating baths in the spinning process to separate out the barium sulfate during spinning and deposit it in the viscose threads in an extremely finely divided state. It has also been proposed to introduce insoluble barium compounds in a finely divided or comminuted state into the viscose to thoroughly permeate it and then to spin the viscose. Experience has shown that there are limitations in a procedure which seeks the thorough or uniform distribution of fine particles and that the spinning process is unfavorably or adversely affected thereby. In both the cases mentioned, the insoluble compounds which give the matt appearance are present in a very finely distributed state. This, as experience has shown, is objectionable or undesirable because the finer the particles, the less is the matt effect and moreover the precipitation is irregular or unreliable. The present invention overcomes the disadvantages just pointed out of the known methods in that the insoluble matt-appearance producing substance is localized in cell-like spaces and not in fine particles dispersed.

In the practice of the process in accordance with the present invention, organic compounds, or salts thereof, with such bases as with sulfuric acid form insoluble sulfates and particularly those of vegetable nature or origin are placed in the viscose solution. However, such organic compounds are not, as such, added to and distributed in the viscose, but they are in the form of solutions in solvents that are not soluble in the viscose, but are emulsified therein, and which either completely, or at least partially, evaporate in the process of manufacture of the artificial silk, and particularly, in the drying thereof.

The organic compound solution must be present in such an amount or proportion as by considerable extent not to be affected by any slight solubility thereof in the viscose.

By way of illustration of the invention, the following examples are given:

Example 1.—In an ordinary viscose solution, there is emulsified a solution in a proportion of 0.8%, that consists of three parts barium oleate and seven parts terpineol. The barium oleate dissolves in the terpineol and remains so dissolved in the viscose even after emulsification. The viscose, after the usual deaeration or air removal is spun in a usual or a well-known way in a bath containing sulfuric acid. During this spinning operation, the barium sulfate and the free oleic acid are separated out and remain incorporated in the finished silk in the spaces previously occupied by the terpineol which, in the treatment was removed by evaporation as by means of steam.

Example 2.—In a normal viscose solution, 1% of a solution of four parts strontium oleate in six parts benzol, to which 0.5 part hydrated cresol (methyl-cyclo-hexanol) is added, is emulsified. The viscose is spun in the same way as before described and the product is finished in a similar way.

In the practice of this invention, preference is given to the fatty acid salts of barium, strontium and calcium, which deposit in the spinning bath barium sulfate, strontium sulfate or calcium sulfate. Numerous other combinations of organic salts can be used in accordance with the nature, or the spirit, of the invention, and suitable organic solvents can be used. Thus barium castor oil dissolved in benzol and strontium oleate dissolved in terpineol may be used. And tetralin may be used as a solvent. By the addition of small quantities of hydrated phenol cresol, etc (for example, methyl-cyclo-hexanol), the solubility of the above mentioned compounds in the organic solvents can be greatly increased. Furthermore, solutions of organic compounds of

metals, such as lead, can be used with volatile solvents, when they deposit or there is separated out an insoluble salt in the spinning bath.

The organic salts added are used in amounts or proportious of from 0.2 to 2% by weight of the viscose.

What is claimed is:—

1. An artificial silk filament having an organic acid of vegetable origin and an insoluble sulfate incorporated cell-like therein.

2. A process of making artificial silk filaments which includes the steps of providing a viscose solution and a solution of a metal salt of a higher fatty acid, the metal being selected from the group consisting of barium, strontium and calcium and the solvent of such compound being insoluble, or only slightly soluble in the viscose solution, forming an emulsion of such solution and the viscose solution, spinning the said emulsion in an inorganic acid bath.

3. A process of making artificial silk filaments from a viscose solution which includes the steps of dissolving in an organic solvent a salt of a higher fatty acid and a metal selected from the group consisting of barium, strontium and calcium, such organic solvent being insoluble or only slightly soluble in the viscose solution, emulsifying the solutions, the salt solution being between 0.2 to 2% of the viscose solution and spinning the emulsion in a sulfuric acid bath, thus forming an insoluble sulfate and a fatty acid compound within the thread.

4. A process as in claim 2 in which the higher fatty acid is of vegetable origin.

5. A process as in claim 3 in which the higher fatty acid is of vegetable origin.

6. A process as in claim 2 in which the viscose solution contains the barium salt of a fatty acid.

7. A proces as in claim 3 in which the viscose solution contains the barium salt of a fatty acid.

8. A process as in claim 3 in which to the organic solvent hydrated phenol is added.

9. A process as in claim 2 in which the viscose solution contains a solution of barium-oleate in terpineol.

10. A process as in claim 2 in which the viscose solution contains a solution of strontium-oleate in benzol to which methyl-cyclo-hexanol is added.

11. An artificial silk filament having incorporated therein an organic acid and an insoluble inorganic salt existing in cell-like spaces within the filament.

12. A process as in claim 2 in which the organic salt is in solution with a volatile solvent and the latter is evaporated to produce cell-like spaces.

THEODOOR KOCH.