Patented July 18, 1939

2,166,741

UNITED STATES PATENT OFFICE

2,166,741

ARTIFICIAL SILK

Hans Karplus, Frankfort-on-the-Main, Germany, assignor, by mesne assignments, to North American Rayon Corporation, a corporation of Delaware

No Drawing. Application November 16, 1927, Serial No. 233,747. In Germany December 8, 1926

20 Claims. (Cl. 18—54)

This invention relates to artificial silk filaments having a dull lustre and to a method for making said filaments. This application is a continuation-in-part of my application Serial No. 132,043 filed August 27, 1926 and resulting in United States Letters Patent No. 1,707,164 dated March 26, 1929.

It has already been proposed for the manufacture of artificial silk of dull lustre, subsequently to impregnate the artificial silk structure, which has been prepared in the usual way, with metal salts, for example barium sulphate, which are difficultly soluble or insoluble in water, in such a manner that the artificial silk is treated successively with aqueous solutions of chemical compounds which by a double chemical reaction yield difficultly soluble or insoluble compounds. The artificial silk of dull lustre obtained in this way has however the disadvantage of an increased specific weight and at the same time a diminished pliability and elasticity.

It has been found that artificial silk having a rich dull lustre, which resembles or is identical with the lustre of natural silk, can be produced by incorporating with the artificial silk filaments difficultly volatile substances, if desired a plurality of such substances in a fine state of subdivision, for instance in such a manner that these substances are distributed in an emulsion-like, suspension-like or colloidal form in the spinning solutions, and these are then worked up according to the usual methods to form filaments. Under difficultly volatile substances are also to be understood substances which are practically non-volatile or generally considered as non-volatile. Such substances may be for example mineral, vegetable or animal oils, fats or waxes, fatty acids, aniline, tetralin, nitro-benzene and the like, and the spinning solutions may be for instance viscose, ammoniacal copper oxide, nitro-cellulose or cellulose acetate solutions. The substances to be incorporated should be so chosen for the different spinning solutions that they do not dissolve molecularly in the spinning solutions, but are merely distributed in the same after the manner of an emulsion or colloid. Solid substances such as for example thorium oxide, magnesium soaps, calcium naphthenate and the like, which are insoluble or difficultly soluble in the spinning solutions may however also be incorporated in a finely suspended or colloidal form with the spinning solutions.

The fine distribution of the added substances in the spinning solution can be obtained for example by preparing these substances beforehand in a very fine state of subdivision or in the form of colloidal solutions and thereupon mixing them with the spinning solutions; however it may also be obtained by purely mechanical means directly in the spinning solutions themselves, for instance by using mixing apparatus, or distributing apparatus. The incorporated substances should preferably be distributed in such manner that, the smaller the diameter of the artificial silk filament to be spun the finer are the distributed particles. For the usual kinds of artificial silk, particles of the size of 0.001 to 0.005 mm. are satisfactory. When distributing apparatus having a more intensive action, for instance homogenising apparatus or emulsifying apparatus are used, it is possible if desired to obtain an even finer distribution of the substances to be incorporated, if necessary up to colloidal dimensions. Mixtures of difficultly volatile with volatile substances, for example paraffine oil and benzene, are also suitable for the present process.

The substances may also be incorporated during the process for the production of the spining solutions, that is to say the substances need not only be distributed in the finished spinning solutions, but they can be successfully distributed beforehand, for instance at the same time as the cellulose compounds are dissolved in their solvents.

The incorporated substances are present in the finished artificial silk structures in the form of microscopic or sub-microscopic drops or granules and give the artificial silk structure a lustre similar to that of natural silk and the textiles made therefrom are extremely pliable and elastic.

According to one mode of carrying out the invention the method adopted is such that the incorporated substances are subsequently partly removed again, by which means it is possible at the same time to obtain the so called hollow filaments. This partial elimination can be obtained for instance by treatment with suitable solvents for example organic solvents. It can also be effected for example by removing a part of the incorporated substances as by heating, if desired, in a vacuum. By this method it is also possible partially to remove more difficultly volatile substances from the filaments again. These artificial silk structures also have a lustre similar to that of natural silk and are characterised by a pleasant soft feel.

Examples

1. 1000 g. viscose, in which 5 g. paraffine oil or linseed oil or mixture of paraffine oil or linseed oil

and small amounts of benzene have been finely distributed by means of a butter churn, is spun in a known manner in precipitating baths, which contain sulphuric acid and sodium sulphate, and the resulting artificial silk filaments washed and dried in the usual way. An artificial silk filament is obtained, which contains tiny drops of oil, distributed very finely and evenly therein, this filament having a rich dull lustre.

2. 150 g. of sodium cellulose prepared and ripened in the usual manner are treated in a known way with 18–26 g. carbon disulphide, 3 g. aniline or paraffine oil then added and after thorough mixing in a kneading apparatus in a known manner dissolved in soda lye, so that a viscose is obtained containing 7–8% caustic soda and 7–8% cellulose. This spinning solution is spun into filaments as in Example 1, which filaments are then washed, dried and thereupon treated with benzene while being heated, until about ⅕ of the incorporated aniline or paraffine oil is extracted from the artificial silk filaments. After evaporating the benzene, artificial silk filaments having a dull lustre are obtained which are particularly soft and pliable.

I claim:

1. A method for making artificial silk filaments having a dull lustre comprising the steps of incorporating foreign substances in a fine and even distribution in the silk filaments and removing a part only of the incorporated foreign substances from the filaments whereby the pitted surface resulting from the said removal contributes to the desired delustering effect.

2. A method for making artificial silk filaments having a dull lustre comprising the steps of spinning in the usual manner solutions of cellulose compounds containing foreign substances in a fine and even distribution and removing a part only of the foreign substances from the filaments obtained whereby the pitted surface resulting from the said removal contributes to the desired delustering effect.

3. A method for making artificial silk filaments having a dull lustre comprising the steps of spinning in the usual manner solutions of cellulose compounds containing mixtures of foreign substances which are difficultly volatile at the normal temperatures in the manufacture of artificial silk filaments and more easily volatile foreign substances in a fine and even distribution and removing a part of the foreign substances from the filaments obtained.

4. A method for making artificial silk filaments having a dull lustre comprising the steps of incorporating in the silk filaments foreign substances in a fine and even distribution and removing a part only of the foreign substances from the filaments by treatment with solvents whereby the pitted surface resulting from said removal contributes to the desired delustering effect.

5. A method for making artificial silk filaments having a dull lustre comprising the steps of incorporating in the silk filaments mixtures of foreign substances which are difficultly volatile at the normal temperatures in the manufacture of artificial silk and more easily volatile foreign substances in a fine and even distribution and treating the filaments with substances, removing a part of the foreign substances from the filaments.

6. A method for making artificial silk filaments having a dull lustre comprising the steps of incorporating in the silk filaments mixtures of foreign substances which are difficultly volatile at the normal temperatures in the manufacture of artificial silk and more easily volatile foreign substances in a fine and even distribution and treating the filaments with substances for removing the easier volatile foreign substances from the filaments.

7. As a new article of manufacture, an artificial silk filament having a dull lustre resembling that of natural silk, said filament containing in a very fine and evenly distributed undissolved form a residual amount of a foreign substance and having a pitted outer surface resulting from a removal of a portion of the foreign substance.

8. An artificial silk filament having a lustre resembling that of natural silk and containing finely divided, dispersed, undissolved particles of one or more substances which provide light interference, said filament also having void spaces therein.

9. An artificial silk filament having a lustre resembling that of natural silk and containing finely divided, dispersed, undissolved particles of oils, said filament also having void spaces therein.

10. An artificial silk filament having a lustre resembling that of natural silk and containing finely divided, dispersed, undissolved particles of tetralin, said filament also having void spaces therein.

11. An artificial silk filament having a lustre resembling that of natural silk and containing finely divided, dispersed, undissolved particles of nitro-benzene, said filament also having void spaces therein.

12. As a new article of manufacture, an artificial silk filament having a dull lustre resembling that of natural silk, said filament containing in a very fine and evenly distributed undissolved form a residual amount of an oil and having a pitted outer surface resulting from a removal of a portion of the oil.

13. As a new article of manufacture, an artificial silk filament having a dull lustre resembling that of natural silk, said filament containing in a very fine and evenly distributed undissolved form a residual amount of tetralin and having a pitted outer surface resulting from a removal of a portion of the tetralin.

14. An article as described in claim 12 in which the size of the finely dispersed particles is from 0.001 to 0.005 mm.

15. A method for making artificial silk filament comprising the step of spinning in the usual manner solutions of cellulose compounds containing mixtures of a substance which is difficultly volatile at the normal temperatures in the manufacture of artificial silk filaments and more easily volatile foreign substances insoluble in the spinning solution in a fine and even distribution, whereby a portion of said substances will be retained and a portion removed to give the finished product a dull lustre.

16. As a new article of manufacture, an artificial silk filament containing an oil in finely dispersed, evenly distributed, undissolved form, said finely dispersed particles being of a size of from 0.001 to 0.005 millimeter.

17. As an article of manufacture, an artificial silk filament containing in finely dispersed, evenly distributed, undissolved form, a substance of the group consisting of mineral oils, vegetable oils, fats and waxes, which substances provide light interference, said article having a portion of the originally added substances removed from the filament to provide a pitted outer surface.

18. A method for making artificial silk fila-

ments having a reduced lustre, comprising the step of incorporating in the silk filaments mixtures of foreign substances which are difficultly volatile at the normal temperatures in the manufacture of artificial silk and more easily volatile foreign substances, in a fine and even distribution, and treating the filaments to remove the major portion of the more easily volatile substance without removal of substantial portions of the more difficultly volatile substance.

19. As a new article of manufacture, an artificial silk filament having a dull lustre resembling that of natural silk, said filament containing in a very fine and evenly distributed, undissolved form a residual amount of nitro-benzene and having a pitted outer surface resulting from a removal of a portion of the nitro-benzene.

20. A method for producing artificial filaments of regenerated cellulose having a dull lustre comprising preparing a spinning solution, adding to the spinning solution at least one substance of the group consisting of mineral oils, vegetable oils, fats and waxes, which substances provide light interference, agitating the spinning solution to form a fine and even distribution thereof, spinning filaments therefrom and removing a portion of the substances distributed in the said filaments to provide a filament having a pitted outer surface.

HANS KARPLUS.