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MANUFACTURE OF ARTIFICIAL SILK

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This invention relates to the production of artificial silk of improved hand and subdued lustre, and contemplates an improved artificial silk of this character and a novel spinning solution from which such artificial silk may be produced. More specifically, the invention is concerned with the incorporation of insoluble alcohols into spinning solutions and into artificial silk filaments produced from these spinning solutions.

As a result of my investigations, I have discovered that artificial silk filaments having a subdued lustre and of excellent hand and suppleness may be produced by incorporating therein insoluble alcohols derived from or constituents of fats and waxes, such as cetyl alcohol, stearyl alcohol, oleyl alcohol, cholesterol, carnaubyl alcohol, or ceryl alcohol. When insoluble alcohols of this character are incorporated into a spinning solution, artificial silk filaments produced from such solutions are exceedingly supple, have a downy touch, and are suitable for immediate fabrication without additional treatment.

Based on this discovery the present invention involves the incorporation and/or dispersion of insoluble alcohols derived from fatty materials into spinning solutions, and spinning artificial silk filaments therefrom with the result that artificial silk of subdued lustre, great suppleness and soft hand are produced.

The term "insoluble" is here used to mean substantially insoluble in water. By "fatty materials" I mean organic fats and waxes.

Among the insoluble alcohols derived from fatty materials which may advantageously be used in the practice of the invention are:

(1) Alcohols of the ethane series such as cetyl alcohol, stearyl alcohol, ceryl alcohol, carnaubyl alcohol, octodecyl alcohol.

(2) Alcohols of unsaturated series such as olefine alcohols and diolefine alcohols; and

(3) Alcohols of the cyclic series, such as zoosterols (cholesterol, ischolesterol, etc.) and phytosterols (sitosterols, brassicasterols, etc.). Alcohols of this cyclic series may be described conveniently as sterols.

The aforementioned insoluble alcohols derived from fatty materials may be incorporated and dispersed in the spinning solution in any suitable manner. They may be warmed if necessary to a temperature above their melting point, and be directly whipped or beaten into the spinning solution by means of a suitable dispersing device, such as a so-called colloid mill. They may be first mixed with a portion of the spinning solu-

tion, and the resulting mixture then added to and mixed with the remainder of the spinning solution. In some cases, it may be convenient to mix the insoluble alcohols with a volatile solvent such as acetone or ethyl ether, the resulting mixture then being incorporated into the spinning solution by mixing.

The insoluble alcohols may also be incorporated into the spinning solution by first mixing them with mineral oil or other oleaginous compound with which they are miscible and thereafter incorporating the mixture into the spinning solution. In the event that mineral oils or other oleaginous materials are mixed with the alcohols prior to incorporation into the spinning solution, it is frequently convenient to add a small amount of stabilizing material, such as sulphonated oil, sulphonated alcohols or related compounds such as stearyl alcohol sodium sulphonate.

Following thorough agitation in order to insure uniformity of dispersion of the insoluble alcohols in the spinning solution, the solution may be spun in the manner customary for the particular variety of the artificial silk being manufactured, employing the customary coagulants.

I attribute the subdued lustre, the increased suppleness and the improved hand of artificial silk produced according to my invention to the presence of fine particles of insoluble alcohols dispersed throughout the filaments. Microscopic examination of the filaments leads me to believe that the fine particles of insoluble alcohols are present in the form of talcky flakes. I believe that these flakes act as internal lubricants within the filaments and thus account for the increased suppleness and softer hand. At the same time, the presence of the flakes serves to change the refractive index of the filaments to light and thus produces the subdued lustre.

When insoluble alcohols are used in conjunction with mineral oil or other oleaginous compound with which they are miscible at elevated temperatures and the resulting mixture is incorporated into filaments of artificial silk through the medium of the spinning solution, the insoluble alcohols apparently precipitate out as minute flakes within the oil globules dispersed through the filaments. I attribute the particularly desirable results obtained in this manner to the effect of a two-phase lubricant within the filament. Thus, a given amount of mixture of mineral oil and alcohol derived from a fatty material produces a great and better effect than several times the amount of mineral oil alone. The action of

the mixture of mineral oil and talcky flakes of precipitated insoluble alcohols may be analogous to the action of multi-phased lubricants, such as the mixtures of graphite and oil used in machinery. The lubricating effect of such multi-phased lubricants appears to be enhanced, that is, the lubricating effect of the mixture is greater than the lubricating effect of its individual components. Whether or not this be the explanation, the fact remains that mixtures of mineral oil and insoluble alcohols impart an increased suppleness and improved hand to artificial silk filaments in which they are incorporated, at the same time accomplishing a marked delustering of the material.

My invention will be more thoroughly understood in the light of the following examples, which illustrate practices of the invention:

Example I

Into 1000 parts of a fresh viscose solution maintained at 18° C. and containing 7% of cellulose, is emulsified 60 parts of a mixture of:

	Parts
Mineral oil of 75 sec. Saybolt viscosity at 100° F.-----	59
Stearyl alcohol-----	40
Stearyl alcohol-sodium sulfonate-----	1

The mixture of mineral oil, stearyl alcohol and stearyl-alcohol sodium sulphonate should be prepared at 50° C. and then cooled rapidly during continuous agitation to 30° C.

The resulting viscose-mineral oil stearyl alcohol emulsion is aged for about sixty hours or until it attains a 10.4 Hottenroth index of ripeness. The emulsion is then spun into a coagulating bath containing 9.5 parts of sulfuric acid and 200 parts sodium sulfate per 1000 parts of water, and filaments are thus produced.

The lustre of the filaments produced in this manner is not only similar to that of natural silk, but after desulfurization and washing, the yarn is naturally soft with a feather-like feel. No special scour of sizing is necessary to render it suitable for fabrication.

Example II

To 1000 parts of a nitrocellulose collodion containing 21% of nitrocellulose are added 10 parts of a white mineral oil of 100 sec. Saybolt viscosity at 100° F., and 10 parts of stearyl alcohol. After thorough mixing in a colloid mill, the mixture is spun, denitrated, and washed to produce regenerated cellulose filaments according to the heretofore customary methods.

The filaments thus produced have a subdued lustre and an increased softness as compared with a yarn which has been delustered with mineral oil alone.

Example III

To 1000 parts of a fresh viscose solution, maintained at 18° C. and containing 7% of cellulose are added 60 parts of a mixture consisting of equal proportions of a white mineral oil of 75 sec. Saybolt viscosity at 100° F. and stearyl alcohol, together with 10 parts of finely divided titanium dioxide. The resulting mixture is thoroughly emulsified and the viscose is spun in a coagulating

bath similar to that used in Example I. The resulting artificial silk filaments are highly delustered, are of superior softness and have a downy hand.

In Example III barium titanate or other suitable pigment may be substituted for titanium dioxide.

The invention may be utilized in the production of any of the customary varieties of artificial silk. In viscose or cupra-ammonium silks, the insoluble alcohol or alcohols are present in the spinning solution in a finely emulsified condition. In acetonic collodion or cellulose acetate or ether-alcohol collodion of nitrocellulose the insoluble alcohol, or a mixture of insoluble alcohols may be in solution. In either case, after spinning the insoluble alcohols are more or less uniformly dispersed as fine plate-like particles throughout the artificial silk filaments.

The heterogeneous dispersion of the mixture of stearyl alcohol and mineral oil (as in the foregoing examples) gives a particularly pleasing artificial silk. The mineral oil employed is preferably white in color, substantially non-acid, having a Saybolt viscosity of from 65 to 100, measured at 100° F.

It will be understood that the examples given herein are intended to be purely illustrative and not restrictive. Changes in procedure will undoubtedly occur to the man skilled in the art, without, however, departing from the basic concept of my invention.

I claim:—

1. An artificial cellulosic silk filament having substantially uniformly dispersed therein mineral oil and flakes of a water insoluble alcohol in such relative proportions that lustre of the filament is reduced and hand and suppleness of the filament are improved, the insoluble alcohol being selected from the group consisting in cetyl alcohol, stearyl alcohol, ceryl alcohol, carnaubyl alcohol, octadecyl alcohol and oleyl alcohol, olefine alcohols, diolefine alcohols, zoosterols and phytosterols.

2. An artificial cellulosic silk filament having substantially uniformly dispersed therein mineral oil and flakes of stearyl alcohol in such relative proportions that lustre of the filament is reduced and hand and suppleness of the filament are improved.

3. An artificial cellulosic silk filament having substantially uniformly dispersed therein mineral oil and flakes of oleyl alcohol in such relative proportions that lustre of the filament is reduced and hand and suppleness of the filament are improved.

4. An artificial cellulosic silk filament having substantially uniformly dispersed therein white mineral oil having a Saybolt viscosity of from 45 to 350 seconds measured at 100° F. and flakes of a water insoluble alcohol in such relative proportions that lustre of the filament is reduced and hand and suppleness of the filament improved, the insoluble alcohol being selected from the group consisting in cetyl alcohol, stearyl alcohol, ceryl alcohol, carnaubyl alcohol, octadecyl alcohol and oleyl alcohol, olefine alcohols, diolefine alcohols, zoosterols and phytosterols.

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