

UNITED STATES PATENT OFFICE

2,234,307

DELUSTERED ARTIFICIAL SILK AND THE
METHOD OF MANUFACTURING SAME

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No Drawing. Application March 8, 1938,
Serial No. 194,599

6 Claims. (Cl. 106—40)

The present invention relates to an improved method for reducing or eliminating the luster of artificial silk and to the improved product obtained thereby.

5 The normal artificial silk fibers are usually as clear as glass and highly lustrous, both of which properties are undesirable for many purposes, such as garments of various kinds. For such purposes, the fiber is made more or less opaque. 10 This is accomplished by mixing inert oils of various kinds into the spinning solution, such oils being dissolved out after the precipitation or solidification of the yarn, whereby the surface of the yarn becomes irregular with resulting 15 reduction in luster. The luster has heretofore also been reduced by adding to the spinning solution a white filling material, such as powdered asbestos, barium sulphate, or titanium dioxide, these added materials rendering the yarn 20 opaque.

The addition of these known filling materials has a number of disadvantages. In the first place, the filling material, especially in the case of the heavy mineral substances, tends to precipitate out in the vessels containing the spinning liquid, and the precipitated material clogs the filter and the spinning or extrusion nozzles. 25 A perhaps more serious objection arises from the fact that these additions represent dead material in the yarn, being of an entirely different character from the cellulosic substance of the yarn and incompatible therewith, and as a result considerably reduce the strength of the yarn, particularly as no natural bond forms between 30 the opacifying or delustering agent and the yarn substance. 35

A further disadvantage which arises in the case of mineral fillers, like barium sulphate and titanium dioxide, is that they increase the average specific gravity of the yarn, and tend to 40 make it brittle and also harsh to the touch.

A still further disadvantage of the use of mineral fillers or of like material, is that such material is not readily capable of absorbing dyes or other material, whereas the type of material 45 contemplated for use by me as the filler readily absorbs dyes and coloring. The product will thus be capable of having imparted thereto a uniform color throughout the silk yarn and 50 filler. The filler can thus be dyed before the dyeing of the silk, and therefore various color effects, combinations of colors in fillers and yarn, may be produced.

55 The present invention is based on the concept that it would be highly desirable to in-

corporate in the artificial silk a delustering filling material having as nearly as possible the properties, aside from luster, of the yarn substance, so as thereby to obtain the desired delustering effect without disadvantageously affecting, at least not to an appreciable extent, 5 the other properties of the yarn, such as its strength, feel, weight, etc.

According to the invention, I employ as an opacifying or delustering filler for artificial silk 10 of diverse origin, a cellulosic material of the proper degree of comminution which has been so modified that it forms a strong union with the artificial silk matrix but is at the same time characterized by the feature that it does not 15 swell in water although it is capable of absorbing water. I thus obtain a delustered artificial silk which retains its strength even in wetting and shows no tendency to swell by reason of the nature of the delustering agent. This cellulosic 20 filler is of approximately the same specific gravity as the artificial silk, so that delustering of the latter can be effected without at the same time "loading" it. Moreover, the cellulosic 25 filler is of approximately the same texture and feel as the artificial silk, resembling the latter in these respects much more closely than the known inorganic fillers. Also, the cellulosic material represents a very cheap filler, and moreover, shows a tendency to form some kind of 30 combination or association with the substance of the artificial fiber, so that it does not exist in the finished fiber, at least not wholly, as separate particles foreign to the substance of the yarn and tending to weaken the latter, but rather as a 35 combined component which even acts to strengthen the yarn.

I have found that a powdered cellulose prepared as described in my copending application, Ser. No. 187,965, filed January 31, 1938, constitutes a superior delustering agent for artificial silk because it possesses to a marked degree the desirable properties above referred to. This powdered cellulose, as described in said application, may be prepared by subjecting cellulose 45 to the action of aqueous formaldehyde in the presence of a strong acid like nitric acid, at elevated temperatures, and with or without simultaneous drying, the product being ultimately dried if it has previously been subjected to 50 the above-mentioned solution at the elevated temperatures but without drying. Cellulose so treated is capable of being easily ground to a fineness of 200 mesh and even finer without hydrating the cellulose. This ground cellulose is 55

unusually strong while wet, does not swell in water, and is more moisture-proof than ordinary cellulose, being in fact quite insensitive to moisture.

5 As described in my application above referred to, the modified cellulose may be prepared by immersing cellulosic material in more or less the naturally occurring cellular condition, such as wood, cotton, ramie, bagasse, etc., for several
10 seconds, in an aqueous bath containing formaldehyde and a small amount of acid, such as a bath consisting of 100 kg. of 40% formaldehyde, $\frac{1}{2}$ kg. of concentrated nitric acid and 50 kg. of water, after which the excess liquid is withdrawn
15 by pressing, centrifuge, or otherwise, and the material then heated to a temperature which is preferably above 100° C. but not so high as to injure the material, and preferably with simultaneous drying. A temperature of the order
20 of 120° C. has been found to be highly satisfactory. If the material is not dried during the heating (during which the reaction on the cellulose occurs) it is then dried and pulverized in any order, the reaction being continued until a
25 highly brittle product is obtained. This material can be pulverized to a condition of fineness resembling starch powder.

The powdered cellulose employed by me does not tend to precipitate from the spinning solution during any stage in the manufacture of
30 the yarn, being of approximately the same specific gravity as the artificial fiber, and therefore does not clog the filters or the nozzles.

In the case of artificial yarn produced by an
35 alkaline process, as in the case of viscose yarn, there occurs a cementing of the cellulose filler particles with the spinning solution during the spinning, resulting in the production of a yarn which is considerably stronger than one produced with known fillers. With non-alkaline
40 spinning solutions, the added cellulosic filling material absorbs the solvent by reason of its porosity and thereby becomes intimately incorporated with the cellulosic substance of the spinning solution during the mixing, extrusion and
45 precipitation or drying. In these non-alkaline processes, there is likewise obtained an artificial silk yarn having a pleasingly subdued luster.

The amount of cellulosic filling material to be
50 employed in any given case will depend largely upon the degree of delustering desired. Ordinarily, an amount of cellulosic filler equal to about 2 to 3% by weight of the artificial silk will be sufficient to reduce the high luster of
55 artificial silk to that of natural silk. The maximum size of the particles may vary within rather wide limits, except they should not reach such a size as would be liable to clog the nozzles which are to be employed with the particular cellu-
60 losic composition. Cellulosic powders having a maximum particle size corresponding to about 200 mesh, or smaller, have been found to give very satisfactory results. In general, the particle size should be of the order of that employed in
65 the case of the known mineral fillers like barium sulphate or titanium dioxide, for example, about 50 to 100 microns.

The cellulosic filling material may be mixed

with the spinning solution in substantially the same way as the known filling materials now employed. Thus, the mixing may take place at room temperature. It may be desirable in some instances to keep the filling material in contact
5 with the spinning solution for a somewhat longer period of time than is the case with mineral fillers, in order to enable the filler to become chemically associated with the spinning solution, but ordinarily, the time normally required
10 for preparing the mixture and extruding and precipitating or drying it, will be sufficient to produce the desired effect.

As already indicated, the cellulosic filler, according to the invention, may be employed with
15 different kinds of artificial silks, including viscose, cellulose acetate, cellulose ether and cellulose nitrate silks. It will be obvious that the cellulosic filler can, if desired, be used, in conjunction with known fillers. Other variations
20 may be resorted to within the scope of the appended claims without departing from the principles of the invention.

Also, the invention is obviously applicable to materials of any cellulosic form, whether artificial silk, or cellulosic products, fibrous or other,
25 for instance, in the form of ribbon, films, etc.

I claim:

1. An artificial silk material of reduced luster, having incorporated therein a ground cellulosic
30 material which has been rendered brittle by heating with aqueous formaldehyde and mineral acid followed by drying and characterized by substantial freedom from swelling on wetting with water.

2. An artificial silk of reduced luster, having incorporated therein a powdered formaldehyde-
35 and acid-pretreated cellulose characterized by substantial freedom from swelling on wetting with water.

3. An artificial silk of reduced luster, having incorporated therein a comminuted cellulosic
40 filler of a maximum particle size of the order of 50 to 100 microns and produced by heating cellulose with formaldehyde and a strong acid to a temperature of about 120° C. followed by
45 drying and grinding.

4. An artificial silk of reduced luster, having incorporated therein about 2 to 3% of powdered
50 cellulose of a maximum particle size of about 100 microns and produced by heating cellulose with formaldehyde and nitric acid to a temperature of about 120° C. followed by drying and grinding in any order.

5. An acetate silk of reduced luster, having incorporated therein a powdered, formaldehyde-
55 and acid-pretreated cellulose characterized by substantial freedom from swelling on wetting with water.

6. Artificial silk fibers having suspended there-
60 in about 2 to 3% of their weight of finely powdered cellulose prepared by heating cellulose fibers with aqueous formaldehyde and a small proportion of mineral acid until a brittle product is obtained, followed by drying and grinding in
65 any order.

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