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TREATED ARTIFICIAL THREAD AND METHOD OF PRODUCING SAME

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6 Claims. (Cl. 8—20)

The present application is in part a continuation of and in part an improvement upon and modification of the disclosure of my copending cases 186,575 filed April 25, 1927 and 367,148 filed May 29, 1929 (now Patents 1,989,098 and 1,989,099, respectively). In said prior cases I have described the treatment of viscose threads (including viscose silk) made by contacting a shaped stream of viscose with a strong mineral acid, e. g. H_2SO_4 of 50% or stronger and preferably H_2SO_4 of 55% or stronger, or other acids of like strength, said threads or silk being generally known in the industry as "Lillienfeld thread" or "Lillienfeld silk". Such products can be made according to the disclosure of my U. S. Patents 1,683,199 (Re-issue 18,170), 1,683,200, 1,820,811, 1,881,740 and 1,881,742, and the corresponding foreign patents. The said threads (silk) are characterized by a dry tenacity exceeding 2 grams per denier, and often 4 to 6 grams per denier and more, but also by a low degree of extensibility, this latter being a drawback which for some time militated against an extensive use of such strong threads. The threads may also have a high luster (as characteristic of viscose silk generally) and a soft silky feel.

In accordance with the disclosure of Ser. No. 186,575, I have found that the extensibility can be substantially increased by treating the Lillienfeld threads with a mercerizing agent such as NaOH solution of 15 to 18% strength or stronger, and allowing shrinking to take place during said treatment.

In Ser. No. 367,148, I have found similar results to be effected by the similar use of NaOH solutions of 2 to 5% strength. In said case it was also stated that NaOH solutions of over 5% and below 15%, although they would cause an increase in extensibility, would also cause a great loss in some of the other useful properties, such as luster, soft silky feel, dry and wet tenacity, etc. It has now been found, and this constitutes the basis of the present case, that NaOH solutions of over 5% and below 10% strength, and particularly NaOH solutions of between about 5.4% and about 9%, are usable in the treatment, and that the decrease in tenacity (wet and dry) is usually not excessive, and the loss in silky feel is sometimes only slight and sometimes negligible, but the loss of luster is sometimes rather great. And with solutions of about 6 to 7% the loss in tenacity and in soft silky feel can be both very low. In fact the loss in tenacity in some cases, when using NaOH solution of about 6-7% may be not substantially greater than the cor-

responding loss when employing NaOH solutions of 18-25% strength.

I call attention also to the fact that the thread (silk) produced on a factory scale is in most cases, injured less by treatment with NaOH solutions of this order of concentration, than is the thread (silk) produced in the laboratory.

While I have above referred to the use of NaOH solutions, it will be understood that other mercerizing agents (especially caustic alkali solutions and alkali metal sulfides) of an equal degree of shrinking activity (and other caustic alkalies of an equal degree of alkalinity) can be used with similar results.

It will be understood that the Lillienfeld threads or Lillienfeld silk, can be treated with mixtures of alkali metal hydroxide and alkali metal sulphide, and that in such cases, and the increase in extensibility can often be produced simultaneously with the desulphurization of the threads. The threads can be treated before or after desulphurization, bleaching and/or drying.

The treatment can be conducted as in the prior cases, by immersion of the threads, advantageously in the form of skeins or hanks, in the alkali solution, while allowing shrinking of the threads to occur. A mild tension can be used but the tension should be let off or reduced during the latter part of the treatment. Preferably tension is omitted. Other methods of contacting the alkaline solution with the threads, such as spraying, running the threads over rollers wet with the solution, dripping the solution on the threads etc., can also be used. The time of contact of the solution with the threads, can vary between wide limits, e. g. a time of a few seconds up to several hours can be used, but it is to be understood that if the thread is not under tension, the amount of shrinkage will be very little after the threads have become thoroughly wetted with the solution. In other words, substantially all the shrinkage occurs immediately on completely wetting the threads with the solution.

The amount of shrinkage varies with different silks, and different concentrations of the shrinking agent. A Lillienfeld silk produced in the laboratory having a given dry tenacity, also may shrink more and may be more injured in dry and wet tenacity, and undergo more loss of luster, when treated with a given concentration of alkali than a commercially produced Lillienfeld silk of the same initial dry tenacity when treated with alkali solution of precisely the same strength.

Thus laboratory made Lillienfeld silk, treated with a 6% NaOH solution, shrunk about 28%

while a similar commercial sample of Lillienfeld silk shrunk only about 17%. A sample of commercially made Lillienfeld silk treated with a 6.4% NaOH solution, shrunk about 10%. Laboratory samples treated with 7%, 8% and 10% NaOH solutions shrunk respectively 31%, 39% and 34%. The four treated laboratory samples lost their luster to a considerable extent. They were reduced in tenacity somewhat, (both wet and dry) and the sample treated with the 10% solution of NaOH, was (after such treatment) only about as good as untreated high quality viscose silk of the ordinary kind.

Three samples of laboratory made Lillienfeld silk, treated respectively with NaOH solutions of 6%, 7% and 10%, showed a great loss of luster in all cases, no considerable loss in softness, the feel of the treated products being good, (no substantial loss of silky feel).

The following table shows some of the measurements of four tests made in accordance with the invention. The Lillienfeld silk, in the form of skeins, was hung in a tall cylindrical vessel holding the NaOH solution at about 15° to 17° C., for about 1 to 5 minutes. Then the skein was lifted out of the alkaline solution and immersed in a 10% H₂SO₄ at 15° C., for 5 minutes. Then the silk was well washed in cold water and dried. No tension was put upon the silk at any part of the treatment. The luster of the commercial sample was not greatly damaged. The luster of the second and third samples was greatly reduced. The luster of the fourth, after treatment, was about like mercerized cotton, (i. e. still greater reduced than the luster of the second and third sample).

Silk treated	NaOH sol.	Dry tenacity		Wet tenacity		Extensibility	
		Before	After	Before	After	Before	After
	Percent					Percent	Percent
Commercial	6.4	5.444	3.872	3.967	2.864	6.5	11.9
Laboratory	6	5.177	3.151	3.473	1.808	4.7	13.2
Laboratory	7	4.353	2.103	2.870	1.857	5.2	17.5
Laboratory	10	4.435	1.625	2.758	0.746	5.7	12.2

Loss of luster, or decrease in luster, is not necessarily an objection. In fact it is known in the art to add pigments, etc. to viscose, before spinning the same in order to make "delustered" (or low luster) artificial silk, since often the very high luster of viscose silk is objectionable. Accordingly the decrease in luster produced in the present process may be in some cases a desirable feature.

Some loss of strength is not necessarily a serious objection to the process. Even the last sample given in the above table, after treatment, is about as good, in wet and dry tenacity, as the best quality of ordinary artificial silk, and this is far better, as to wet and dry tenacity, than most of the ordinary viscose silk on the market.

I prefer, in the present invention, to use NaOH solutions of about 5.4 to 7%.

As in my copending cases noted above, neutral salts, alkaline salts, organic materials, known in the mercerizing industry, can also be added to the shrinking solutions. Examples of such materials are sodium salts (chloride, nitrate, sulphate, silicate, etc.) and the corresponding potassium salts, glycerine, alcohol, sugars, etc.

The process is applicable to filaments, fibers, yarns, threads, woven, knitted or netted fabrics composed of the same. Also mixed goods can be similarly treated, (any silk, wool or other animal fibers or yarns thereof being preferably first

coated with albumen, starch, gelatin or other protective sizing). In the claims, the term "threads" is intended to cover such materials.

The following example is given, for the purpose of illustration.

Factory made viscose silk, made according to the process of Example I of my Reissue Patent 18,170, in the form of previously moistened skeins, is introduced, without tension, into a deep bath of aqueous solution of NaOH, of 6.4% strength, at ordinary temperature, say 15 to 20° C. After standing for about 2 minutes, the skeins are raised from the liquor, allowed to drain for about one minute, and then lowered into a deep bath of 10% H₂SO₄, allowed to stand for 5 minutes, then transferred to a washer in which the silk is well washed with cold or hot water, and dried. No tension is applied during the process. The extensibility is increased without any considerable loss of dry and wet tenacity or silky feel. The luster is slightly lowered.

The details of the operation can vary between wide limits.

I have referred above to the use of the shrinking agent in a cold state. It is in no way necessary that the said solution be used cold. It can be used warm or hot. But if used at or near boiling temperature, it is advisable to limit the time of contact of the threads with the liquor, to about 5-10 minutes, as a longer treatment, hot, might cause more injury to the tenacity. In ordinary cases, I prefer to use the solution cold, which is less troublesome, it not being necessary to heat the liquors. It was rather surprising to find that alkaline shrinking agents could be used

cold, in view of the fact that in some of my early work on increasing extensibility of Lillienfeld silk, it was found that cold solutions were more injurious to said silk than warm or hot solutions, (cf. Ser. 367,150 now Patent 1,989,101).

The difference in behaviour between the factory made Lillienfeld silk referred to above on the one hand and the laboratory made Lillienfeld silk on the other, is probably due in large part at least, to the fact that the factory spinning machinery gives a more regular stretching than the laboratory machinery.

I claim:—

1. Process of treating commercially produced viscose threads having a dry tenacity substantially exceeding 2 grams per denier but a low extensibility, which comprises subjecting said threads to the action of a caustic alkali solution having a concentration above 5% but below 10%, calculated as NaOH, while allowing shrinkage of the threads to occur whereby the extensibility of said threads is substantially increased.

2. Process of treating factory made viscose threads having a dry tenacity substantially exceeding 2 grams per denier but a low extensibility, which comprises subjecting said threads to the action of a caustic alkali solution having a concentration between about 5.4% and about 9%, calculated as NaOH, while allowing shrinkage of the

threads to occur whereby the extensibility of said threads is substantially increased.

5 3. Process of treating factory made viscose threads having a dry tenacity substantially exceeding 2 grams per denier but a low extensibility, which comprises subjecting said threads to the
10 the action of a caustic alkali solution having a concentration between about 6% and about 7%, calculated as NaOH, while allowing shrinkage of
15 of said threads to occur whereby the extensibility of said threads is substantially increased.

4. Process of treating factory made Lillienfeld silk, which comprises subjecting said threads to the action of a caustic alkali solution having a
15 concentration above 5% but below 10%, calculated as NaOH, while allowing shrinkage of the threads to occur whereby the extensibility of said threads is substantially increased.

20 5. As a new product, artificial threads produced from viscose, said threads having a dry

tenacity not below 2 grams per denier, and an extensibility at least 8%, and having a reduced degree of luster as compared with ordinary factory made Lillienfeld silk, which product can be
5 produced by treating factory made Lillienfeld silk with caustic alkali solution of about 5.4% to about 9% concentration, calculated as NaOH, and allowing permanent shrinking of the same to occur during said treatment.

6. A process of treating Lillienfeld silk made on
10 a commercial scale, which comprises subjecting the same to the action of a shrinking agent which has a shrinking action about equal to that of NaOH solution of over 5% but below 10% strength, and which does not have a substantially greater
15 deleterious action on the dry and wet tenacity and soft silky feel, than NaOH solution of said concentration, while allowing shrinkage of the threads to occur.

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