

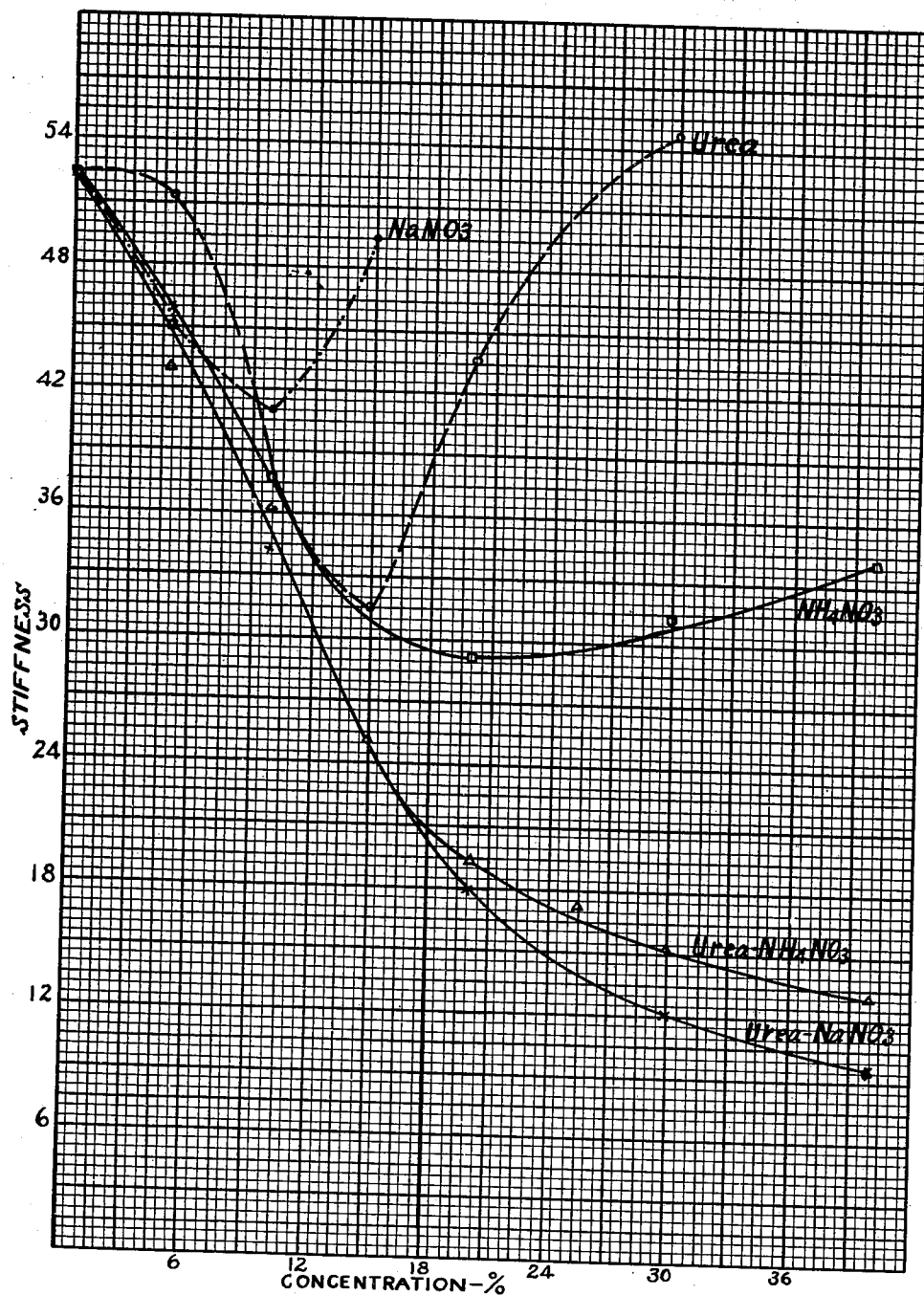
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TREATMENT OF SILK

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EFFECT OF SALTS AND UREA ON SILK

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## UNITED STATES PATENT OFFICE

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## TREATMENT OF SILK

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This invention relates to the preparation of silk for fabrication. It consists in a process that may be performed upon the raw silk or upon the yarn in the course of or after the completion of the throwing operation. The object in view is the avoidance of the necessity of wetting the yarn when it is about to be fed to fabricating machinery—to knitting machinery for the manufacture of full-fashioned hosiery, for example—and so avoiding the complications and the ill effects of such wetting.

The accompanying drawing presents a plotting of values and a tracing of curves upon coordinate paper, in illustration of the ensuing description.

In the course of the throwing operation as commonly conducted, the raw silk is soaked in an emulsion of oil. The oil used may be a vegetable oil or a mineral oil, or a sulfated oil, or a combination of these. Emulsifying agents are commonly used, such as soaps, sulfated alcohols, and sulfonated compounds. The emulsions may also contain alkali, penetrants, and hygroscopic agents, such as glycerol and the glycols. Small amounts of salts are sometimes added. After the silk, treated with some combination of these materials, has been doubled, twisted, steamed, and coned, the finished yarn is ready for fabrication, as in knitting machinery. It may, however, be further conditioned by storage under high humidity. In any case, however, in procedure heretofore known, the thread must be wetted before fabrication; and, to this end, the yarn, in its advance to the knitting machine, is caused to pass through a trough containing water or an aqueous solution.

The invention proceeds from the discovery that urea has softening and plasticizing effect upon the sericin of raw silk; that is to say, upon the outer envelope that surrounds the strands of fibroin.

I have found that when raw silk is treated with molten urea, the sericin is very rapidly dissolved and removed, while the fibroin remains unchanged. Utilization of this solvent action to produce a softening or plasticizing effect is limited by the fact that urea is solid at ordinary temperatures and, being in solution, tends to crystallize out of the urea-sericin system. The presence of such crystals would be injurious to the physical properties of the individual silk fibers.

I have found that, by the addition of certain salts, and bringing them together with the urea into solution (typically aqueous solution), crys-

tallization may be prevented, and by this means the softening effect may be greatly increased. In such case, using the solution of the strength and in the quantity hereinafter specified, the sericin is not removed, as it is in the high-temperature treatment in molten urea alluded to above, but, remaining, has become softened, with the consequent good effect here specified. No chemical change in the sericin is apparent, and so far as is known none occurs.

The urea-salt mixtures may be introduced into the silk fiber most conveniently by soaking the silk in their water solutions and allowing the water to dry off.

The degree to which the silk has been softened may be determined by a measurement of its stiffness. The following data were obtained by adaptation of a Gurley stiffness tester (used to determine the stiffness of paper or cloth) to this material. The values given represent the stiffness of groups of eight hundred fibers arranged in the form of brushes, which were about three-fourths of an inch wide. The silk was soaked in the solution for one hour and then tested after exposure to standard atmosphere for twenty-four hours.

*Stiffness of silk soaked in various solutions*

| Concentration of solution, per cent | Urea | NH <sub>4</sub> NO <sub>3</sub> | NaNO <sub>3</sub> | Urea 47%, NH <sub>4</sub> NO <sub>3</sub> 53% | Urea 70%, NaNO <sub>3</sub> 30% |
|-------------------------------------|------|---------------------------------|-------------------|-----------------------------------------------|---------------------------------|
| 0                                   | 52.5 | 52.5                            | 52.5              | 52.5                                          | 52.5                            |
| 5                                   | 51.5 | 45.2                            | 45.0              | 42.8                                          | 45.8                            |
| 10                                  | 37.9 | 37.9                            | 41.0              | 36.1                                          | 34.8                            |
| 15                                  | 31.5 |                                 |                   | 25.2                                          |                                 |
| 20                                  | 43.7 | 29.2                            | 50.0              | 19.4                                          | 19.4                            |
| 30                                  | 55.3 | 31.1                            | 62.0              | 15.6                                          | 12                              |
| 40                                  |      | 34.0                            |                   | 13.1                                          | 9.6                             |

These data serve to show that much greater softening may be effected by the use of the urea-salt mixtures than by use of urea alone. When more than fifteen per cent of urea alone is present, a network of crystals forms in the fiber, and the stiffness is increased.

Referring to the drawing, the figures indicative of the stiffness of samples of silk after treatment with solutions of urea alone, of salt (nitrate of sodium, nitrate of ammonium) alone, and of urea and salt together, and at varying degree of concentration, are plotted. And from this plotting it appears that, whereas urea alone and salt alone have effect, at low degrees of concentration, in reducing stiffness, at higher degrees of concentration the effect is reversed and stiffness in-

creases again. It further appears that urea and salt together have effect in reducing stiffness that is progressive throughout the range of concentration explored (up to 40%) and that the maximum effect of the urea-sodium nitrate solution exceeds that of the simple solution of urea alone in inverse ratio of 9.6:31.5.

I have found that yarn prepared from silk treated with associated urea and salts may be successfully thrown and knitted if a sufficient quantity of the mixture is used to cause a decrease of 60 per cent or more in the stiffness of the silk fibers. A content of twenty to thirty per cent of the urea-salt mixture, taken up by the silk, is necessary to produce this softening effect.

The permissible ratios of urea to salt are those which prevent the crystallization of both urea and salt from the urea-salt-sericin system. The exact amount of the mixture and the ratio of urea to salt to be used will vary with the nature of the salt and the degree of softening desired.

The salt that I employ in association with urea is a salt of one of the alkali metals, or of ammonium, or a blend of such salts. In the ensuing claims I shall use the term alkali-metal salts as inclusive of ammonium salts. Certain salts of the alkaline earth metals, e. g., calcium nitrate, may be used. Their use is, however, relatively disadvantageous, since their presence interferes in the boiling-off and finishing processes.

The specific salts that I find preferable are the nitrates of ammonium and of sodium and the acetate of sodium. These preferences are based upon economic as well as upon technical considerations. Certain salts, such as potassium acetate, while giving good results, are undesirable because of cost, while others, such as sodium chloride, are cheap but do not give so good a result as the preferred salts. I have found no alkali-metal salt that will not afford the effect described.

As an example, 100 pounds of skein silk may be soaked in an emulsion containing: water, 375 pounds; soluble oil, 7 pounds; sodium bicarbonate and sodium carbonate, 1 pound each; soap, 1 pound; urea, 87.5 pounds; and sodium nitrate, 35 pounds. The emulsion is warmed to 35° C., and the silk is allowed to soak for one or two hours. The silk is then taken from the bath and the excess solution is removed by some suitable means, such as centrifuging. The skeins are then dried and thrown into yarn.

The oils and the soap are removed almost in entirety from the soaking bath and taken up by the silk. The urea and salt, however, are removed and taken up by the silk to the extent that the solvent (water) is taken up. Ordinarily the silk absorbs an amount of the solution equal to its own weight, and the amount of the urea-salt mixture that is so taken up by the silk is in accord with the degree of concentration in which the mixture is carried in solution in the soaking bath. That is to say, if the solution be, for example, a twenty-five percent solution, and if the weight of silk be 100 pounds, 100 pounds of solution will be taken up, and this solution will carry 25 pounds of the solute. A portion of the alkali is neutralized by the natural acidity of the silk.

The excess solution may be recovered by suitable means and re-used, after addition of oils and soap in the proper quantities and adjustment of the alkali content.

It is not necessary that the silk be treated in the skein. Silk that has been soaked in the usual manner may be treated with the urea solution of my invention at any point in the throwing operation or in the form of yarn when otherwise ready for fabrication.

While it is not necessary, it ordinarily will be desirable, in the practice of the invention, that the soaking bath contain the usual oils, as well as the urea-salt mixture.

The amounts of urea and of salt will vary from those of the example given above, to afford softening to the desired degree.

The urea and associated salt taken up by the silk as described and with softening and plasticizing effect, so that wetting is not requisite to the preparation of the silk for fabrication, will in the further processing of the fabricated article be removed.

It has been found that silk treated in the manner described above is not only capable of being knitted without wetting, but does not ordinarily require any setting of the twist. An additional advantage is the greater ease with which the sericin may be removed.

I claim as my invention:

1. The method herein described of preparing silk for fabrication by the softening of the sericin of the fibroin-surrounding envelope, which consists in subjecting the silk fibre, prior to the fabrication of the yarn, to a solution of urea together with a salt of a group that consists of the nitrates and acetates of the alkali metals.

2. The method herein described of preparing silk for fabrication by the softening of the sericin of the fibroin-surrounding envelope, which consists in subjecting the silk fibre, prior to the fabrication of the yarn, to a solution of urea together with sodium nitrate.

3. Silk yarn prepared for fabrication and carrying a 20-30% burden of a non-crystallizing association of urea and a salt of a group that consists of the nitrates and acetates of the alkali metals.

4. The method herein described of preparing silk for fabrication by the softening of the sericin of the fibroin-surrounding envelope, which consists in subjecting the silk fibre, prior to the fabrication of the yarn, to a solution of urea together with ammonium nitrate.

5. The method herein described of preparing silk for fabrication by the softening of the sericin of the fibroin-surrounding envelope, which consists in subjecting the silk fibre, prior to the fabrication of the yarn, to a solution of urea together with sodium acetate.

6. Silk yarn prepared for fabrication and carrying a 20-30% burden of a non-crystallizing association of urea and sodium nitrate.

7. Silk yarn prepared for fabrication and carrying a 20-30% burden of a non-crystallizing association of urea and ammonium nitrate.

8. Silk yarn prepared for fabrication and carrying a 20-30% burden of a non-crystallizing association of urea and sodium acetate.

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