

UNITED STATES PATENT OFFICE.

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SILK PRODUCT AND PROCESS FOR THE TREATMENT OF SILK.

No. 931,433.

Specification of Letters Patent.

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To all whom it may concern:

Be it known that I, JACOB KNUP, a citizen of the United States, and resident of Paterson, in the county of Passaic, State of New Jersey, have invented a certain new and useful Silk Product and Process for the Treatment of Silk, of which the following is a full, clear, and exact specification.

My invention relates to a process of treating silk to produce therefrom a weighted silk of a uniform blue black color and to the product of the said process.

One of the objects of the invention is to provide a process of producing a weighted silk of a uniform blue black color which will be particularly simple, inexpensive and efficacious as compared with processes heretofore employed and a second object of my invention is to provide a tin-weighted silk having a uniform blue black color and which, however heavily weighted, is stronger and generally more satisfactory than weighted blue black silks heretofore produced.

In the practice of my process, I first weight the silk and directly thereafter subject it to a bath of pyrolignite of iron (black-iron) or its equivalent, then to a bath containing tannin. The treatment of the silk with a tannin-containing bath must follow and not precede the treatment with pyrolignite of iron or its equivalent.

The weighting treatment is preferably that employed to weight silk which is to be dyed in colors or is not to be dyed black and is hereinafter set forth. The dyeing treatment hereinafter termed "blue black dye treatment" is substantially the treatment to which unweighted silk is subjected to dye the same blue black. This treatment is also hereinafter set forth.

In the weighting treatment as preferably practiced, a solution of chlorid of tin preferably tin tetra-chlorid, is used in combination with a solution of phosphate of soda, either alone or with a solution of silicate of soda. Instead of using the phosphate of soda or the phosphate and silicate of soda, I may employ a solution of ammonia, soda or potash, or a carbonate of one of these, the alkalies being, it is to be understood, the equivalents of the phosphate or phosphate and silicate of soda in the practice of my process.

In the preferred practice of the weighting

process, the silk is first boiled off and is thereafter put into a cold solution of tin tetra-chlorid of about 30° Baumé and subjected to this solution for approximately an hour, after which the silk is washed and then worked in a warm solution of phosphate of soda or other alkali of 4° Baumé at a temperature of about 150° F. for approximately one hour. It is then again washed and thereafter subjected to a warm solution of silicate of soda of 3° Baumé at a temperature of about 120° F. for about one hour. The silk is then again washed and the treatment repeated until the desired weight is obtained. Instead of following this particular order of treatment, the silk may, as hereinbefore indicated, be alternately subjected a number of times to the solutions of chlorid of tin and phosphate of soda and after the last treatment in either the tin or phosphate baths, the silk may be advantageously subjected to a solution of silicate of soda. It is immaterial after which solution the weighting is terminated, whether after subjecting the silk to the tin or the phosphate bath, and after subjecting the silk to the weighting treatment as described, it may be advantageously subjected to the action of an acid or alkaline bath. After the silk has been weighted in the manner set forth, it is next subjected to the "blue black dye treatment" which consists in first subjecting the weighted silk to a solution of pyrolignite of iron, *i. e.*, a solution of iron in crude acetic acid and then to a solution of tannin-containing material.

In practice, I employ a bath of iron pyrolignite solution of 1° to 5° Baumé, but the strength of the solution depends upon the shade desired, the stronger the solution, the bluer will be the shade resulting from the treatment. The silk is worked in the solution of iron pyrolignite at a temperature of approximately 60° F., for about one hour and is then wrung out and after being covered for about two hours, is worked and is then placed in the tannin-containing bath. This bath is preferably composed of a solution of gambier, or rather tannin-containing material to which has been added extracts of logwood and fustic or other yellow vegetable dye, the proportions of which ingredients vary according to the shade desired, and the silk is worked in this solution for approximately two hours. It has been found that

good results are obtained from a solution of about 100 pounds of gambier to 100 pounds of silk, the solution having added thereto, about 20 pounds of logwood extract, and 40 pounds of fustic.

During the last described operation, which is continued for about two hours, the solution is preferably maintained at a temperature of about 160° F.

After the treatment in the tannin-containing bath, the solution is worked and subjected to a solution of logwood extract and soap, and thereafter the silk is finished by treatment with lemon juice and oil. The bath of logwood extract and soap preferably contains about 20 pounds of logwood extract and 50 pounds of soap to each 100 pounds of silk treated, and the silk is preferably allowed to remain in this solution for about two hours, the temperature being raised from about 110° F., at the outset until the desired blue color is obtained. This usually occurs at a temperature of about 150° F., although the results vary somewhat according to the character of the silk under treatment and the difference in working in the weighting process.

In the final step of the process, I preferably employ a solution of approximately two gallons of lemon juice and one and one-half pounds of oil, preferably olive oil, to each hundred pounds of silk treated.

The product of the hereinbefore recited process may be identified and recognized by its behavior upon being subjected to the usual burning and acid tests, which behavior may be readily determined by one skilled in the art by applying such tests to the said product. Upon immersion in concentrated oxalic acid, the product produced as hereinbefore set forth, it will be found to turn a reddish brown or light yellow and, upon burning, such product will leave a well preserved ash structure having a slightly brownish or salmon shade.

Having now described my invention what I claim as new and desire to secure by Letters Patent is:

1. The process for the treatment of silk to obtain a weighted silk of a uniform blue black color, which consists in weighting the silk, then subjecting it to a bath of pyrolignite of iron and thereafter to a bath containing tannin.

2. The process for the treatment of silk to obtain a weighted silk of a uniform blue-black color, which consists in weighting the silk with tin, a phosphate and a silicate; then subjecting the silk to a bath containing iron and then to a bath containing tannin and logwood, after the iron bath but not before.

3. The process for the treatment of silk to obtain a blue-black color, which consists in weighting the silk with tin, a phosphate and

a silicate; then subjecting the silk to a bath of pyrolignite of iron and then to a bath containing tannin and logwood.

4. The process for the treatment of silk to obtain a blue-black color, which consists in weighting the silk with successive baths containing a salt of tin, a phosphate and a silicate; then subjecting the silk to a bath of pyrolignite of iron, then subjecting the silk to treatment with tannin-containing material and logwood, and then to a bath with logwood and soap.

5. The process for the treatment of silk to obtain a blue-black color, which consists in weighting the silk in successive baths containing tetra chlorid of tin, phosphate of soda and a silicate; then subjecting the silk to a bath of pyrolignite of iron, and then to treatment with gambier, logwood, and fustic, and then to a bath with logwood and soap.

6. The process for the treatment of silk to obtain a blue-black color, which consists in weighting the silk in successive baths containing tetrachlorid of tin, phosphate of soda and a silicate; then subjecting the silk to a bath of pyrolignite of iron, then to treatment with gambier, logwood and fustic, then to a bath with logwood and soap, and lastly to treatment with an acid and oil.

7. The process of dyeing tin-weighted silk a uniform blue-black, which consists in weighting the silk with tin, then subjecting it to a bath of black iron, and finally treating it with suitable vegetable extracts or dyes.

8. The process of dyeing silk blue-black, obtaining a product of any desired weight; which consists in weighting the silk with tin, subjecting it in sequence to an acidulated bath, a black-iron bath, a black-iron gambier bath, containing matter for coloring according to the shade desired, and, then, to the dye bath, substantially as described.

9. A weighted silk of a uniform blue black color, said silk being impregnated with a tin salt and pyrolignite of iron and said silk turning a reddish brown or yellow shade upon immersion in concentrated oxalic acid and leaving a well preserved ash structure upon being burned, said ash structure having a slight brownish or salmon color.

10. A weighted silk of a uniform blue black color, said silk being impregnated with a tin salt, a phosphate, a silicate and pyrolignite of iron tannin and a vegetable dye and said silk turning a reddish brown or yellow shade upon immersion in concentrated oxalic acid and leaving a well preserved ash structure upon being burned said ash structure having a slight brownish or salmon color.

11. A weighted silk of a uniform blue black color, said silk being impregnated

with a tin salt, a phosphate and pyrolignite of iron.

12. A weighted silk having a uniform blue black color, said silk being impregnated
5 with a tin salt, a phosphate, a silicate, and pyrolignite of iron.

13. A weighted silk having a uniform blue black color, said silk being impregnated

with a tin salt, a phosphate, a silicate, pyrolignite of iron and tannin.

In witness whereof I have hereunto set my
hand in presence of two witnesses. 10

JACOB KNUP.

Witnesses:

GLEN ELOISE McELHONE,
OTTO MUNK.