

UNITED STATES PATENT OFFICE.

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TREATMENT OF MATERIALS CONTAINING SILK.

935,292.

Specification of Letters Patent.

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No Drawing.

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To all whom it may concern:

Be it known that we, THEODORE BOETTGER and FREDERICH VOM EYSER, citizens of the United States, the said THEODORE BOETTGER residing at Hackensack, Bergen county, New Jersey, and the said FREDERICH VOM EYSER residing at Rochelle Park, Bergen county, New Jersey, have invented certain new and useful Improvements in the Treatment of Materials Containing Silk, of which the following is a specification.

Our invention relates to the treatment of materials composed wholly or in part of raw silk in order to prepare it for subsequent dyeing and other operations usual in the art, and its novelty consists in the manner in which the materials are treated and the reagents employed in the treatment.

Raw silk consists chiefly of two substances: The true silk fiber which is called fibroin, and which comprises about two-thirds or more of its substance, and an outer layer, or covering, of a material called in the art "bast" and scientifically known as sericin, which makes up the bulk of the remainder. It also contains a very small amount of wax, fat, coloring matter and ash. Most of the coloring matter is in the outer sericin layer. Sericin is a substance much resembling gelatin in its properties and is soluble in water only by prolonged boiling. Fibroin is a proteid and is not noticeably affected by prolonged boiling in water, but is somewhat readily attacked by caustic alkalies, even in weak solutions, their action rendering it more brittle and rough and diminishing its gloss. Fibroin is also attacked by soap solution, if it is boiled for a long time therein, but it is not acted upon by weak acid solutions.

In the preparation of silk for the dye bath, it has been customary to first treat it in a manner which is termed "boiling off" which consists in boiling it in a bath of soap and water, sometimes with the addition of carbonate of soda. The purpose of this treatment by boiling off is to remove the outer layer of sericin whereby the silk becomes lighter in color, since most of the coloring matter is contained in the sericin layer, and the luster is developed and it becomes softer and more suitable for dyeing. During the process of boiling off, the sericin first swells up making the silk sticky. It

then dissolves leaving the lustrous and internal thread exposed. In treating piece goods, which are composed in part of cotton or wool, the boiling off process serves the further purpose of cleansing from the material whatever dust may be adhering there- to and it tends also to improve the quality of the cotton or wool mixture. It is also customary to put the goods through a washing process after boiling off.

The boiling off and washing process consume much time and labor and employ materials which, while not expensive in themselves or in small quantities, become expensive when used in large quantities as they must be used in the customary practice of the art.

The purpose of our invention is practically to eliminate the boiling off and washing process. To that end we treat the materials with a solution, in a novel way and with the result that not only is an enormous economy effected but the goods are stronger, they are less liable to injury in the subsequent treatment to which they are subjected, they dye more rapidly, the finished product has a better appearance and they can be stripped after dyeing without deterioration in quality.

We first prepare our solution which consists essentially of a sulfated organic oil, water and an alkaline reagent sufficient in quantity to render the liquid alkaline in reaction but insufficient injuriously to affect the fibrous material of the goods. By "sulfated organic oil" we mean an animal or vegetable oil, for instance, castor oil, which has been subjected to the action of sulfuric acid within certain limited ranges of temperature washed with a salt solution and then treated with an alkali partly to neutralize the acid reaction. The object of this treatment is to render the oil miscible in water. A good example of such a sulfated organic oil is the liquid called "Turkey red oil". A certain amount of unchanged oil and salts of the fatty acids of the oil and various derivatives of the constituents of the oil are formed in the course of the treatment outlined and are to be found with the oil. This oil and others of the same class produced in substantially the same way, are not suitable for our purpose. They are usually acid in reaction to litmus even after the addition of a small portion of an alkali. In the

preparation of our solution we add to 1 liter of water, about .75 cc. of the sulfated organic oil, .075 cc. of ammonia and from .75 to 1.5 grammes of sodium carbonate. Of course it will be understood that these proportions are relative. They are those which we prefer in the light of our present knowledge. This solution it will be noted is in effect a weak solution slightly alkaline in character of the sulfated oil in water. It comprises substantially one part of the oil to 1300 parts of water. The proportion of water may be decreased possibly to 200 parts without essentially varying the process but such concentration is not necessary and when we say a "weak solution" we mean one in which the water is present in a greater proportion than 200 parts and yet one strong enough to accomplish our purpose. Persons skilled in the art will readily understand the limits of economy and efficiency for any particular kind of oil and alkali. Assuming this solution to have been prepared with the ingredients in the proportion stated, the raw silk or mixed goods is passed through the bath, which is previously brought to a temperature of not quite 100° C. and are allowed to remain in contact with the solution for a space of about twenty minutes.

The sulfated oil seems to impart a high capillarity to the materials treated causing the liquid to penetrate the fibers thoroughly and efficiently. The solution also seems to have unusual and superior dissolving and emulsifying properties. The weak alkaline substance seems to act primarily as a hydrolyzing agent upon the sericin and incidentally it seems to increase the solvent power of the solution. The solution seems moreover to be very mild in its action upon the fibroin and leaves it coated with a very thin layer of nitrogeous material which is repellant to water, though soluble, on prolonged boiling therein, or if an alkaline reagent is added thereto. This layer probably protects the fibroin from weakening, not only during the time that it is in the bath, but also during the subsequent operation of dyeing. This later is removed in great measure during the subsequent treatment of the goods. Fibroin coated with such a thin layer of sericin, we believe to be an entirely novel product or composition of matter.

It will be understood that other weak alkaline substances may be used in place of sodium carbonate in connection with the sulfated oil, and that such substances must be used in a quantity sufficient to render the solution alkaline in reaction. The rate of removal of the sericin seems to vary with different alkaline reagents. Of the substance which we have tested, for instance, borax, was found to be quite efficient. Disodium hydrophosphate is less efficient than the borax if used in equal amount and di-

sodium ammonium phosphate less efficient yet. Ammonia alone might be used, but a comparatively large proportion would be necessary. It is preferable to use an alkaline salt of an oxygenated acid because its action is moderate and it does not disastrously affect the fibroin.

Of course, it will be understood that if, on account of previous or subsequent operations in the treatment of the silk, the substitution of other alkaline reagents for the sodium carbonate is deemed desirable, then the precise quantities to be employed and the time during which the material should be subjected to their influence, must in each instance be determined by previous experiment, in a manner well known to those skilled in the art. After treatment with this solution the materials are ready immediately for the dyeing operation and can be placed in the dye bath without washing.

If the exigencies of manufacture are such that the materials are wholly or partly dried after treatment in our novel solution then it is sometimes preferable for instance, when skein goods are treated, to wet them out before dyeing. Under other circumstances, however, for instance, with mixed silk and cotton goods, they may be wholly dried and dyed without wetting or washing. As an example including the step of dyeing, we will describe the dyeing of Canton floss a light blue color. The silk is placed on sticks in usual manner and kept moving in a bath consisting of

Water	1000.00 parts.
Turkey red oil	1.00 "
Ammonia	.10 "
Soda Ash	1.00 "

at a temperature of about 99 degrees centigrade (just below a simmer), till the desired degree of degumming is attained (in practice about 70 minutes). It is then wrung, wet out, immersed, and moved in a bath consisting of water 750 parts, spent liquor from the boiling off operation 250 parts, which has been made slightly acid by the addition of a little sulfuric acid, and xylene blue 1/10 part, and kept moving till the shade is even and of the desired intensity. (About 35 minutes). It is then taken out, slightly scoured and finished with a little soda-olive oil emulsion. The temperature of the dye bath is about 80 degrees centigrade. Of course the details of the operation may be widely varied without departing from the invention.

What we claim as new is:—

1. The process of treating material containing silk which consists in subjecting it to the action of a hot weak slightly alkaline solution of sulfated oil in water.

2. The process of treating silk which consists in subjecting it to the action of a weakly alkaline solution of sulfated oil in

water at normal temperatures, and second, heating the solution and the silk to a temperature not exceeding that of boiling water.

5 3. The process of treating silk, which consists in subjecting it to the action of a weakly alkaline solution of Turkey red oil in a relatively large quantity of hot water.

10 4. The process of treating silk which consists in subjecting it to the action of a weak solution of Turkey red oil in water rendered slightly alkaline by the addition of an alkaline salt of an oxygenated acid.

15 5. A process of dyeing materials containing silk which consists in subjecting them to the action of a hot weak alkaline solution of sulfated oil in water and subsequently placing them in a dye bath without washing.

20 6. A process of dyeing materials containing silk which consists in subjecting them to the action of a hot weak alkaline solution of sulfated oil in water, drying the material

and subsequently placing them in a dye bath without washing.

7. A process of dyeing materials containing silk which consists in subjecting them to the action of a hot weak alkaline solution of sulfated oil in water, drying the materials, wetting them with water and then placing them in a dye bath without washing. 25

8. A process of dyeing materials containing silk which consists in subjecting them to the action of a hot weak alkaline solution of sulfated oil in water, removing them from the solution, wetting them out with water and then placing them in a dye bath without washing. 30

Witness our hands this 18th day of November 1908, at New York, N. Y.

THEODORE BOETTGER.
FREDERICH VOM EYSER.

Witnesses:

HERMAN MEYER,
WILLIAM R. BAIRD.