

UNITED STATES PATENT OFFICE

CAMILLE DREYFUS, OF NEW YORK, N. Y., AND HERBERT PLATT, OF CUMBERLAND, MARYLAND, ASSIGNORS TO CELANESE CORPORATION OF AMERICA, A CORPORATION OF DELAWARE

PROCESS FOR PRESERVING THE LUSTER OF ORGANIC DERIVATIVES OF CELLULOSE

No Drawing.

Application filed December 30, 1926. Serial No. 153,127.

This invention relates to a new and improved process for the preservation or protection of the luster of artificial silk prepared from organic derivatives of cellulose. More specifically it relates to the treatment of cellulose acetate silk with certain salts so that the silk will retain its luster although it is subjected to a boiling treatment.

It is well known that one of the characteristics of fabrics, filaments etc., prepared from some organic derivatives of cellulose is the fact that such material loses its luster when boiled in water or subjected to a boiling treatment such as is commonly encountered in the dyeing, bleaching and similar processes. This characteristic has somewhat limited the application of such fibers, fabrics, etc., particularly when employed in conjunction with cotton, wool, real silk, etc. In the dyeing of cotton and wool it is usually necessary that the dye bath be maintained at or near a boiling temperature and for this reason great difficulty was encountered in the dyeing of mixtures of cotton and/or wool with cellulose acetate silk because of the consequent delustering of the cellulose acetate silk. The same difficulty is encountered when it is desired to degum real silk in a mixture of real silk and cellulose acetate silk, since this degumming is usually carried on at boiling temperature.

The object of this invention is to devise a process for the prevention, total or partial, of the delustering of artificial silk prepared from organic derivatives of cellulose, whether the same be in thread, fiber or fabric form. Another object of this invention is to prevent the delustering of such artificial silk when in admixture with cotton and/or wool and/or real silk. Another object of this invention is to regulate the amount of delustering which occurs when artificial silk prepared from organic derivatives of cellulose is treated in solutions at or near boiling temperatures.

The applicants have discovered that if artificial silk made from cellulose acetate, cellulose propionate, ethyl cellulose etc., is treated at boiling temperatures in the presence of certain salts, the presence of such salts will serve to completely protect the luster of such arti-

cial silk or lessen the amount of delustering which occurs, depending upon the salt and the amount of such salt present in the boiling bath. The salts which they have found to be most efficient in obtaining these results are the sulphocyanides of the alkali and alkali-earth metals.

Sodium sulphocyanide, potassium sulphocyanide, ammonium sulphocyanide, calcium sulphocyanide, barium sulphocyanide have been utilized with varying degrees of success. The choice of the particular salt to be used will depend upon the nature of the bath in which it is desired to use the same. If the salt is to be added to a dye bath in large quantities it will be necessary to add the sodium or potassium sulphocyanide, preferably the former, and similarly when the salt is added to a bath containing soap. At other times the use of calcium sulphocyanide is recommended. The following are some examples of the manner in which the applicants' invention can be utilized:

Example I

A cellulose acetate fabric was treated at the boil for $\frac{3}{4}$ of an hour in a bath containing 10 grams per litre of calcium sulphocyanide. The fabric, after drying, was found to have lost but very little of its original luster. The fabric after this treatment was found to be denser and softer than the original untreated fabric. A cellulose acetate fabric treated with water at the boil as above in the absence of calcium sulphocyanide was completely delustered.

Example II

A cellulose acetate fabric was subjected to a process as outlined in Example I, using 100 grams of calcium sulphocyanide per litre of bath. The fabric, after drying, had its original luster.

Example III

A fabric of cellulose acetate silk was treated as outlined in Example I, substituting ammonium sulphocyanide for the calcium sulphocyanide. The luster was partly protected by the ammonium sulphocyanide but the pro-

tection obtained was not so good as the protection obtained using calcium sulphocyanide.

Example IV

5 A process similar to that outlined in Example II, employing ammonium sulphocyanide in the place of calcium sulphocyanide, was carried out on cellulose acetate fabric. The
10 luster of the fabric was protected but not to the same extent as in Example II.

Example V

15 A cellulose acetate fabric was treated as in Example I, using 265 grams of ammonium sulphocyanide per litre. The luster of the fabric after this treatment was found to be virtually unaltered.

Example VI

20 A cellulose acetate fabric was treated according to the process outlined in Example I, utilizing 10 grams of sodium sulphocyanide per litre in the place of calcium sulphocyanide. The luster of the fabric, after this
25 treatment, was preserved, but to a lesser degree than it was by the treatments outlined in Examples I and III.

Example VII

30 A cellulose acetate fabric was treated according to the process outlined in Example I, utilizing 10 grams of potassium sulphocyanide per litre in place of calcium salt. The
35 luster of the fabric treated was preserved to about the same extent as in Example VI.

Example VIII

40 A cellulose acetate fabric was treated in a dye bath containing 3% SRA blue III and to which bath had been added 10 grams per litre of sodium sulphocyanide. The fabric was dyed at the boil for one hour. A marked
45 loss in the luster of the fabric was noticed but the relustering was not as great as in a similar treatment in the absence of sodium sulphocyanide. This treatment was repeated in the presence of soap, and it was observed that
50 the fabric suffered a greater loss of luster in the presence of the soap. This indicates that the protective effect of the sulphocyanide is lessened by the presence of soap.

Example IX

55 A cellulose acetate fabric was treated in a dye bath containing 3% SRA blue III and to which bath had been added 50 grams per litre of sodium sulphocyanide. The fabric
60 was dyed at 100° C. for one hour. There was a loss of luster in the fabric but it was not as great as that resulting from the process outlined in the preceding example and employing but 10 grams per litre of sodium
65 sulphocyanide. The hand and appearance of

the fabric was superior to that of similar untreated material.

Example X

Stockings made from cellulose acetate silk were treated in an open tub at the boil for one hour in a bath containing 10 grams of sulphocyanide per litre. The stockings retained their original luster, had a good full handle and did not show any teathy effect.

Example XI

Hosiery made from cellulose acetate silk was treated in an open tub at the boil for one hour in a bath containing 8 grams of sodium sulphocyanide per litre. The stockings thus treated were very slightly delustered and had a good full handle. In addition the stockings did not show any teathy effect such as usually results when stockings are treated according to this or the preceding example, but in the absence of sulphocyanides. It will, therefore, be seen that the use of sulphocyanides, in addition to its protective influence on the luster, results in the prevention of
90 teathiness.

From the above examples it will, therefore, be noted that fabrics made from cellulose acetate and other organic esters and ethers of cellulose can be treated at boiling temperatures without losing their characteristic luster if there is added to the boiling solution any of the various salts enumerated. By varying the salt and quantity of the salt added it is possible to regulate the extent of the delustering. We have found that concentrations of the salt solution used of from 0.1% to 30% are very suitable for this purpose. It is thus possible to obtain the various gradations between a virtually complete delustering and a retention of the normal luster of the fabric.

Of the various sulphocyanides utilized in the above examples the calcium sulphocyanide appears to be the most efficacious in preventing the loss of luster in the organic derivatives of cellulose. The ammonium and barium sulphocyanides are also very good but are not quite as effective as the calcium salt. The sodium and potassium sulphocyanides can also be utilized, being generally slightly less effective than equal amounts of ammonium and barium sulphocyanides. The ammonium, potassium and sodium salts can be used in dye baths to better advantage than the other sulphocyanides.

With reference to the conditions of delustering it can be stated that the amount of delustering which takes place will vary with the conditions and material being treated. Thus a fabric which is being treated on a jig does not deluster easily whereas the same fabric treated on a winch or reel will deluster much more easily and most easily if immersed completely in a water or soap and

water bath at the boil. The ease with which various fabrics will be delustered depends upon the type and construction of the fabric. Generally speaking yarn will deluster more easily than knitted fabric and knitted fabric in turn will deluster more readily than woven fabric. As a general rule the more dense the fabric or its individual yarns the more difficult it is to deluster the same.

In view of the above the examples given herein are to be taken as examples only and will have to be somewhat modified in utilizing the different machines or different types of fabrics. Where it is found that the luster is not sufficiently protected, sufficient protection can be obtained by increasing, within certain limits, the concentration of the sulphocyanide in the bath.

The great advantage of the processes outlined herein over current practice lies in the fact that the luster of organic derivatives of cellulose such as cellulose acetate etc., can be preserved, partially or totally, by employing as little as one to two percent of calcium sulphocyanide or one to five percent of sodium or potassium sulphocyanide. Before this invention if it was desired to treat cellulose acetate and similar materials in boiling solutions it was necessary to add large quantities of salt such as Glauber's salt, sodium chloride, etc., the amounts exceeding 25% in many cases. The presence of these large amounts of salts in the dye bath is troublesome since, among other things, there is a tendency for the dye stuff to be salted out. It is clear that this draw-back does not exist if the applicants' process is utilized since the concentration of the salt rarely exceeds 5%.

By virtue of the ability to regulate, as a result of the applicants' process, the amount of delustering which takes place it is possible to obtain cellulose acetate and similar fabrics which very closely resemble natural silk. This resemblance is very marked not only in the luster but also in the softness and handle of the thus treated fabrics, a feature which has long been desired.

The salts of the classes above specified in addition to having the property of preserving the luster of cellulose acetate and similar artificial silks, have a further desirable effect on such materials. When used in proper concentrations they act as swelling or jelling agents for this class of yarns and fabrics. The improvement in the softness and handle and appearance of the fabric when treated with a solution of the salts is probably due to this swelling action of the salts upon the fabric.

The applicants' invention is not to be considered as limited by the examples hereinbefore given. The invention is of great importance in the treatment of mixed fabrics made from yarns or threads of organic cellulose esters and cotton, wool or silk or any of

them. As heretofore stated any one or more of the salts enumerated can be utilized in this process, the choice of the particular salt being determined by treatment to which the fabric is being subjected. In addition the quantity of the particular salt added can be varied within a wide range depending upon the extent of delustering of the organic cellulose derivative desired. The percentages as given in the claims are based upon the bath and not upon the fabric or material being treated.

Having described our invention what we claim and desire to secure by Letters Patent is:

1. A process of controlling the delustering of materials comprising organic derivatives of cellulose which consists in adding to a hot bath in which such delustering is apt to occur, a salt of the formula $R-CNS$, where R denotes ammonium or a metal of the alkaline or alkaline-earth series.

2. A process of protecting the luster of materials comprising organic derivatives of cellulose which consists in adding to a hot bath in which delustering of such material is apt to occur, a sulphocyanide of an alkali-earth metal.

3. A process of protecting the luster of materials comprising organic derivatives of cellulose which consists in adding to a hot bath in which delustering of such material is apt to occur, sulphocyanide of calcium.

4. A process of preserving the luster of materials comprising organic esters of cellulose which consists in adding to a hot bath in which delustering of such material is apt to occur, a salt of the formula $R-CNS$, where R denotes ammonium or a metal of the alkaline or alkaline-earth series.

5. A process of controlling the delustering of materials comprising organic esters of cellulose which consists in adding to a hot bath in which such delustering is apt to occur, a sulphocyanide of an alkali-earth metal.

6. A process of controlling the delustering of materials comprising organic esters of cellulose which consists in adding to a hot bath in which such delustering is apt to occur, sulphocyanide of calcium.

7. A process of preserving the luster of materials containing cellulose acetate which comprises adding to a hot bath in which delustering is apt to occur, a salt of the formula $R-CNS$, where R denotes ammonium or a metal of the alkaline or alkaline-earth series.

8. A process of controlling the delustering of materials containing cellulose acetate which comprises adding to a hot bath in which such delustering is apt to occur, a sulphocyanide of an alkali-earth metal.

9. A process of protecting the luster of materials containing cellulose acetate which comprises adding to a hot bath in which de-

lustering of such material is apt to occur, sulphocyanide of calicum.

10. A process of controlling the delustering of filaments and fabrics or organic esters of cellulose which comprises adding to a hot bath in which such delustering is apt to occur 0.1%-30% of a salt having the formula R—CNS, where R denotes ammonium or a metal of the alkaline or alkaline-earth series.

11. A process of protecting the luster of filaments and fabrics of organic esters of cellulose which comprises adding to a hot bath in which delustering of such material is apt to occur 0.1%-10% of a sulphocyanide of an alkali-earth metal.

12. A process of preserving the luster of filaments and fabrics of organic esters of cellulose which comprises adding to a hot bath in which delustering of such material is apt to occur 0.1%-10% of calcium sulphocyanide.

13. A process of controlling the delustering of filaments and fabrics of organic esters of cellulose which comprises adding to a hot bath in which such delustering is apt to occur 1% of a salt having the formula R—CNS, where R denotes ammonium or a metal of the alkaline or alkaline-earth series.

14. A process of protecting the luster of filaments and fabrics of organic esters of cellulose which comprises adding to a hot bath in which delustering of such material is apt to occur, 1% of a sulphocyanide of an alkali-earth metal.

15. A process of preserving the luster of filaments and fabrics of organic esters of cellulose which comprises adding to a hot bath in which delustering of such material is apt to occur, 1% of calcium sulphocyanide.

16. In a process for the dyeing of mixed fabrics containing organic esters of cellulose at elevated temperatures the step of adding to the dye bath a salt of the formula R—CNS, where R denotes ammonium or a metal of the alkaline or alkaline-earth series, whereby the luster of the organic derivative of cellulose is preserved.

17. In a process for the dyeing of mixed fabrics containing organic esters of cellulose at elevated temperatures the step of adding to the dye bath a sulphocyanide of an alkali-earth metal, whereby the luster of the organic derivative of cellulose is protected.

18. In a process for the dyeing of mixed fabrics containing organic esters of cellulose at elevated temperatures the step of adding to the dye bath calcium sulphocyanide, whereby the luster of the organic derivative of cellulose is preserved.

19. In a process for the dyeing of mixed fabrics containing cellulose acetate at elevated temperatures the step of adding to the dye bath calcium sulphocyanide, whereby the luster of the organic derivative of cellulose is protected.

20. In a process for the dyeing of mixed fabrics containing cellulose acetate at elevated temperatures the step of adding to the dye bath 1% calcium sulphocyanide, whereby the luster of the organic derivative of cellulose is preserved.

In testimony whereof, they have hereunto subscribed their names.

CAMILLE DREYFUS.
HERBERT PLATT.