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COLORED CELLULOSE MATERIAL

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4 Claims. (Cl. 18—54)

This application is divisional of my application Ser. No. 680,897, filed July 17, 1933.

In the said U. S. Patent application Ser. No. 680,897 there is described a process for direct production of dyed regenerated cellulose material (viscose) consisting in incorporating into the viscous mass in any phase previous to coagulation the solution of an ester-salt of a leuco vat dyestuff, in performing the coagulation and finally in developing the dyeing in the regenerated cellulose material by acid oxidation.

As described in said application Serial No. 680,897 this process allows also the production of special dyeing effects.

It has now been found that in the same way also copper silk, obtained by dissolving cellulose by means of a solution of ammoniacal copper oxide and coagulating the solution with the aid of water or diluted acids or alkalies, whereby cellulose is re-formed, may be directly dyed. According to the present invention a solution of an ester-salt of leuco vat dyestuff is added to the ammoniacal copper oxide cellulose solution as a rule again in any phase of the process previous to coagulation.

Like in the case of viscose, a completely homogeneous mass is thus obtained, in which no precipitations or decompositions take place, which might adversely affect the subsequent spinning process and further operations. This mass is then spun in the ordinary way and brought to coagulation. The produced threads or materials are then subjected to the usual after-treatment for copper silk.

Development of the dyeing takes place by means of acid oxidation during coagulation, or by a suitable after-treatment of the regenerated cellulose after coagulation. As cupric salts in the presence of an acid may act as oxidizing agents, in many cases the copper salt, i. e. the cupric sulphate formed in the coagulation bath is sufficient to develop the dyeing (see U. S. Patent No. 1,917,101). In this case a further oxidative acid treatment is superfluous.

The same effects as in the case of viscose may generally also be attained with copper silk.

According to the present process it is also possible to apply on any kind of fabric an ammoniacal copperoxide cellulose mass to which an ester-salt has been added, to bring the mass to coagulation, and to develop the dyeing therein by means of acid oxidation during or after coagulation. In such a way particular effects are obtained, for example delustred or shiny relied effects, which are dyed fast.

The invention is illustrated, but not limited by the following examples:

Example 1

1 gr. of the ester-salt of leuco diamethoxylibenzanthrone is dissolved in 50 ccm. of lukewarm water and 20 ccm. of ammonia of 25%. 20 ccm. of this solution are thoroughly mixed with 20 ccm. of an ammoniacal copper oxide cellulose solution. This mixture is spun and coagulated at 30° C. in a bath containing 40% of sulphuric acid. The cellulose is regenerated. The copper sulphate formed in the acid coagulation bath acts as oxidizing agent and the green dyeing is developed during the coagulation process. The thus obtained threads are subjected to the usual after-treatment for copper silk.

Example 2

1 gr. of the ester-salt of leuco dibenzpyrenequinone is dissolved in 50 ccm. of hot water. After cooling down, 20 ccm. of ammonia of 25% are added. 20 ccm. of this solution are thoroughly mixed with 20 ccm. of a concentrated ammoniacal copper oxide cellulose solution. The homogeneous mass is spun as usual and the threads are coagulated at 40° C. in a bath containing 30% of caustic soda lye and if necessary small quantities of sugar, glucose, glycerine or any other suitable ingredients. The threads are thoroughly rinsed in water containing 20 grs. of sulphuric acid of 66° Bé. per litre. The ester-salt is not dissolved away from the fibre, neither in this stage nor in the following coagulation bath. Finally the dyeing is developed by treating the material in a bath containing 36 grs. of sulphuric acid of 66° Bé., 3 grs. of sodium nitrite, also 10 grs. of a solution of ammonium vanadate 1:1000 per litre at a temperature of 20–40° C., or in a bath containing 0.5 gr. of sodium bichromate or potassium bichromate, 36 grs. of sulphuric acid of 66° Bé., 20 grs. of a solution of ammonium vanadate 1:1000 and 2 grs. of oxalic acid per litre. Thus one obtains artificial silk threads of a bright, fast yellow.

What I claim is:—

1. A process for manufacturing colored cellulose material, which consists in adding to an ammoniacal copperoxide cellulose solution in any phase previous to coagulation a solution of an ester-salt of a leuco vat dyestuff, performing the coagulation in known manner and developing the dyeing in the regenerated cellulose by acid oxidation.

2. A process for manufacturing colored cel-

lulose material, which consists in adding to an ammoniacal copperoxide cellulose solution an ester-salt of a leuco vat dyestuff, performing the coagulation in a known manner and developing the dyeing in the regenerated cellulose by treating the latter in a bath containing bichromate and acid.

3. A process for manufacturing colored cellulose material, which consists in adding to an ammoniacal copperoxide cellulose solution previous to coagulation a solution of an ester-salt of a leuco vat dyestuff, performing coagulation of the mass by pressing same into a bath containing sulphuric acid, whereby owing to the formation of coppersulphate acting in presence of acid as oxidinz agent, simultaneously with the coagulation the development of the dyeing in the regenerated cellulose takes place.

4. The herein described colored cellulose material obtained by adding to an ammoniacal copperoxide cellulose solution a solution of an ester-salt of a leuco vat dyestuff, performing coagulation in a known manner and developing the dyeing in the regenereated cellulose by acid oxidation.

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