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MANUFACTURE OF SHAPED ARTICLES FROM VISCOSE

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9 Claims. (Cl. 18—54)

Our present invention relates to the manufacture of artificial products of an improved quality from viscose, such as artificial silk, horse hair, ribbons, sheets, capsules and the like, by spinning a viscose solution into an acid salt solution, or coagulating shaped articles in such a bath, and more particularly to the use of a viscose which contains xanthates of aromatic, hydroaromatic, cyclic or heterocyclic alcohols.

When using a viscose having an insufficient degree of maturity or a viscose prepared from cotton linters or from a pulp having a high content of alpha-cellulose and a low content of hemicelluloses, or using a precipitating bath with a high content of salts or a precipitation bath containing a strongly and rapidly coagulating salt, for instance, $ZnSO_4$ or $MgSO_4$, there is the danger that the products obtained, when inspected under reflected or transmitted light, have a turbid or a milky or opaque appearance and, therefore, will be of inferior technical value.

According to this invention we have found that these drawbacks are overcome, and glossy and clear artificial silk and sheets are obtained by employing a viscose which contains ester salts of alkali xanthates of aromatic, hydroaromatic, cyclic or heterocyclic alcohols. Substances of this kind which have proved useful for the purpose in question are, for instance, the sodium xanthates of borneol, of mentol, of fenchyl alcohol, of furfuryl alcohol, of naphthene alcohol, of dekahydronaphthol, of methylcyclohexanol, of hexahydroisothymol, of phenylethyl alcohol and of phenylpropyl alcohol, and the like. These additions are efficacious even when applied in a relatively small amount; thus, for instance the addition of 1 to 5 parts of the sodium salt of the xanthogenic ester acids to 1000 parts of viscose will be sufficient to produce an anti-turbid action which suffices for nearly any required condition of coagulation. The xanthates of the alcohols of the naphthene, terpene and the camphor series are particularly efficient for this purpose, and allow of a wide latitude in the conditions of treatment. Xanthic acid esters of benzyl alcohol and cyclohexanol have but little efficacy, whereas the phenylethylalcohol and the methylcyclohexanol respectively, enriched by a further CH_2 group, gives xanthates of quite a good efficacy. The ester acids formed by reaction with the acid of the precipitating bath are not decomposed when the thread passes through the bath, and are nearly insoluble therein.

The essence of the present invention will more clearly result from the following comparative experiments which have been carried out under extreme working conditions. The examples are not intended to limit the invention to any special spinning conditions, to the kind and the amount of the additions, or to the stage in which the addition is made in the course of the preparation of the viscose.

The following examples illustrate the invention:

Example 1.—When spinning a normal viscose prepared from an alkali cellulose ripened about three days at a temperature of about 15° C. and containing 7½ to 8 per cent. of cellulose and 6½ to 7 per cent. of alkali and having a common salt number of 7½ to 8, into a bath containing 29% of Na_2SO_4, 13% of H_2SO_4 and a small amount of $ZnSO_4$ according to the temperature, the capillary titer, the immersion length in the precipitating bath, the stretch which has been applied and the remaining spinning conditions, a silk is obtained of which all (or at least a great part) of the hanks produced shows an irregular gloss and turbid stripes after having been completely finished. However, when adding to the same viscose an amount of 0.20% of the sodium xanthate of phenyl ethyl alcohol and spinning under exactly the same conditions, a silk is obtained of which all the hanks are unobjectionable. A viscose having a common salt number of 7½ designates a viscose which just coagulates when brought in a solution of common salt of 7½ per cent, but not yet in a common salt solution of 7¼ per cent. strength.

Example 2.—A viscose of a high degree of viscosity prepared from unripened alkali cellulose and containing 6 to 6½% of cellulose and 6½% of alkali is spun into a precipitating bath containing 27% of Na_2SO_4, 3% of $ZnSO_4$, and 11% of H_2SO_4, yielding at any degre of maturity that practically enters into consideration, a mat silk or silk of mean appearance. However, when adding to the viscose 0.25% of fenchyl xanthate and spinning at a maturity of 5½, a glossy silk is obtained.

Example 3.—A viscose of a high degree of viscosity prepared from cotton linters and containing about 7½% of cellulose and 6½ of alkali and having an addition of 0.3% of borneol xanthate is spun, at a common salt number of 5½, into a bath containing 18% of $MgSO_4$, 12% of Na_2SO_4 and 13% of H_2SO_4. In contradistinction with a silk obtained by spinning the same viscose without addition of borneol xanthate, the silk spun from the viscose containing borneol xanthate has a normal and glossy appearance.

Example 4.—A viscose according to Example 1, but having a common salt number of 5 and containing 0,4% of borneol xanthate is spread on a glass plate, and coagulated, while on this support, in a bath containing 15% of $MgSO_4$, 15% of Na_2SO_4 and 12% of H_2SO_4 at a temperature of 30 to 35° C. Whereas a viscose without the said addition only yields turbid and nearly opaque products after desulfurizing the sheets obtained from the viscose containing borneol xanthate are glass-clear.

What we claim is:

1. In the process of manufacturing artificial products from viscose the steps which comprise adding to the viscose the xanthate of a compound belonging to the group consisting of aromatic, hydroaromatic, cyclic and heterocyclic alcohols, extruding the viscose through suitably shaped openings, and coagulating said extruded viscose by means of an acid salt solution.

2. In the process of manufacturing artificial products from viscose the steps which comprise adding to the viscose the xanthate of an aromatic alcohol containing a side chain, extruding the viscose through suitably shaped openings, and coagulating said extruded viscose by means of an acid salt solution.

3. In the process of manufacturing artificial products from viscose the steps which comprise adding to the viscose the xanthate of a compound selected from the group consisting of the camphor series and terpene series.

4. In the process of manufacturing artificial products from viscose the steps which comprise adding to the viscose the sodium xanthate of phenylethyl alcohol extruding the viscose through suitably shaped openings, and coagulating said extruded viscose by means of an acid salt solution.

5. In the process of manufacturing artificial products from viscose the steps which comprise adding to the viscose fenchyl xanthate and coagulating said viscose by means of an acid salt solution.

6. In the process of manufacturing artificial products from viscose the steps which comprise adding to the viscose borneol xanthate extruding the viscose through suitably shaped openings, and coagulating said extruded viscose by means of an acid salt solution.

7. In the process of manufacturing artificial products from viscose the steps which comprise adding to the viscose 0,2% of the sodium xanthate of phenylethyl alcohol extruding the viscose through suitably shaped openings, and coagulating said extruded viscose by means of a precipitating bath containing 13% of sulfuric acid, 29% of sodium sulfate and a small amount of zinc sulfate.

8. In the process of manufacturing artificial products from viscose the steps which comprise adding to the viscose 0,25% of fenchyl xanthate extruding the viscose through suitably shaped openings, and coagulating said extruded viscose by means of a precipitating bath, 11% of sulfuric acid, 27% of sodium sulfate and 3% of zinc sulphate.

9. In the process of manufacturing artificial products from viscose the steps which comprise adding to the viscose 0,4% of borneol xanthate extruding the viscose through suitably shaped openings and coagulating said extruded viscose by means of a precipitating bath containing 12% of sulfuric acid, 15% of magnesium sulfate and 15% of sodium sulfate.

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