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PROCESS OF PRODUCING RAYON

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The present invention relates to a novel process for the maintenance of ideal spinning conditions in the manufacture of rayon, and it relates specifically to the prevention of spinneret incrustations when a viscose solution is extruded into an acid spin bath to form filaments, threads, yarns, ribbons and the like.

In the commercial production of viscose rayon it is necessary for reasons of economy to provide for uninterrupted spinning of even denier filaments. Interruption of spinning is generally caused by the contamination of the spinnerets themselves due to various impurities in the viscose solution and/or the spin bath. These impurities have a tendency to agglomerate or deposit on the spinneret and thus to close gradually its fine orifices. Zinc rings, calcium rings, sulphur craters, precipitated cellulose, resin particles, secondary reaction products, etc., are impurities which cause spinneret incrustations. The spinnerets are especially susceptible to incrustation when acid spin baths are used containing dissolved zinc salts such as for example zinc sulphate. Sulphuric acid is conventionally used in viscose spin baths. Even if the deposit of impurities is insufficient to entirely close the spinneret orifices, it causes variations in the denier and cross sections of the filaments which in turn impart undesirable properties to the finished viscose products. Also, so-called spinning hooks tend to form at the obstructions, either in or around the orifices of the spinneret, which, when filaments are extruded therethrough, will tend to cause a temporary interruption of the spinning of a filament at the orifice affected, thus occasioning tearing of the filament. These deficiencies necessitate frequent and expensive spinneret changes and cause an increase in lower quality yarns.

Although incrustation of spinnerets is especially noticeable when metal spinnerets made from gold, gold-platinum, gold-palladium, platinum-rhodium, tantalum, or any other corrosion-resistant metal, are used, this deficiency is also experienced with spinnerets manufactured from artificial ruby and other semi-precious stones. The prevention of incrustation is becoming more and more important with the increasing tendency in the art to spin finer denier filaments which necessitates the use of smaller orifices.

I am well aware that it has, heretofore, been proposed to prevent the clogging of spinneret orifices by adding cation-active compounds to the spin bath or viscose solution (vide U. S. Patent No. 2,125,031 to Polak and Weeldenburg

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issued on July 26, 1938, and U. S. Patent No. 2,132,929 to Bley issued on October 11, 1938), or by coating the spinnerets with wax-like materials (vide U. S. Patent No. 2,100,581 to Weeldenburg issued on November 30, 1937). By extensive experiments I have unexpectedly found that the incrustation of spinneret orifices is substantially prevented by water-soluble polymerization products of alkylene oxides or substitution products thereof which products become cation-active in acid spinning baths used in the coagulation of viscose thread. In order to be operative they must have at least a limited solubility and stability in acid spin baths and/or viscose solutions.

While I have found that under certain optimum conditions extremely small quantities of these polymers effect the desired result when added to the spin bath, the viscose solution or both, and that 0.0001% of such compounds has been found sufficient, generally I have ascertained that the addition thereof in a considerable excess of 0.05% should be avoided for reasons of economy. I prefer to use an addition of about 0.005% based on the active ingredient of the chosen compounds. Although these polymerized alkylene oxides, etc., may be added either to the spin bath or the spinning solutions, it is preferred that they be added to the spin baths, for the reason that it is now common practice to regenerate spin baths and thereby avoid losses of the constituents thereof.

Polymerized alkylene oxides, adapted to be used in combination with viscose solutions and/or acid spin baths, may be prepared in accordance with processes set forth in British Patent No. 346,550, German Patent No. 597,496, and French Patent No. 750,520, issued to I. G. Farbenindustrie A. G. The degree of polymerization of alkylene oxides may be varied to a large extent by varying the conditions under which polymerization is induced, and I have found that polymers of alkylene and ethylene oxides having a degree of polymerization above 30 are especially useful for preventing spinneret incrustations provided they are sufficiently soluble and stable in spin baths and/or viscose solutions. The alkylene oxides adapted to be polymerized have the structure

in which R represents hydrogen, an alkyl, a cycloalkyl or an aryl radical.

The following examples will serve to illustrate my present invention:

Example I

A conventional viscose solution was spun into a well-known acid spin bath containing sulphuric acid, sodium sulphate, magnesium sulphate and a small quantity of zinc sulphate. When about 0.005% of a polymerized ethylene oxide having a polymerization degree of about 91 (manufactured under the trade name "Wax 4000" by Carbide and Carbon Chemicals Corp., New York, N. Y.) was added, calculated on the spin bath, most of the irregularities and difficulties in spinning were eliminated during a long run.

Example II

The surface-active compound, set forth in Example I, was added to a conventional viscose solution before spinning, and it inhibited the formation of incrustation of the spinneret through which the solution was extended to form viscose rayon during a long run.

Example III

The surface-active compound set forth in Example I was added to both the spin bath and the viscose solution and it inhibited the formation of incrustation on the spinnerets for a relatively long period of time.

Examples of other suitable polymerized alkylene oxides are: polyethylene oxide having a polymerization degree of about 34 (manufactured under the trade name "Carbowax 1500" by Carbide and Carbon Chemicals Corp., New York, N. Y.), polymerized propylene oxides, nonaethylene glycol, etc.

The viscose solution to be extruded through the orifices of a spinneret may contain additional compounds such as delustrants, proteins (casein), etc. Since in the spinning of high strength yarns, i. e., yarns having a dry strength of at least 2 grams per denier, such as those used in tire constructions and mechanical rubber goods (belts, hose, etc.) and in the well-known "continuous" spinning processes, the contamination of the spinnerets is particularly serious, this invention is of special importance in this field. I have, furthermore, found by experimentation that polymerized alkylene oxides, for example, polyethylene oxides which are not only sufficiently soluble and stable in acid spin baths but which in addition are capable of precipitating negatively charged colloidal particles occurring as contaminations in the spin bath, such as for example, colloidal sulphur particles prevent the clogging of spinneret orifices more effectively than alkylene oxides which do not precipitate negatively charged particles. Modifications of my process will readily be recognized by those skilled in the art, and I desire to include all such modifications and variations coming within the scope of the appended claims. In these claims the term "alkylene oxide" is intended to cover ethylene oxide per se, its homologues (for example, propylene oxide, etc.) and substitution products thereof.

I claim:

1. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a spinneret into an acid precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

2. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a corrosion-resistant metal spinneret into an acid precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable surface-active polymerized ethylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

3. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a platinum alloy spinneret into an acid precipitating bath to form filaments and the like therefrom the step which comprises spinning the viscose filaments in the presence of a small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

4. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a spinneret into an acid, zinc-containing precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

5. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a corrosion-resistant metal spinneret into an acid, zinc-containing precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

6. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a platinum alloy spinneret into an acid, zinc-containing precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

7. In the manufacture of a viscose yarn wherein a viscose solution is extruded through minute orifices of a spinneret into an acid, zinc sulphate-containing precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

8. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a corrosion-resistant metal spinneret into an acid, zinc sulphate-containing precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide, whereby

incrustation of the spinneret during the spinning operation is substantially inhibited.

9. A viscose spin bath, for coagulating viscose filaments therein containing a small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide.

10. A viscose spin bath, for coagulating viscose filaments therein, containing a dissolved zinc salt and a small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide.

11. A viscose spin bath, for coagulating viscose filaments therein, containing zinc sulphate and a small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide.

12. A viscose solution containing a small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide.

13. A viscose solution containing about 0.0001 to 0.05% by weight of a dissolved, substantially stable, surface-active polymerized ethylene oxide.

14. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a spinneret into an acid precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable, surface-active polymerized alkylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

15. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a corrosion-resistant metal spinneret into an acid precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable, surface-active polymerized alkylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

16. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a platinum alloy spinneret into an acid precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a small amount of a dissolved, substantially stable, surface-active polymerized alkylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

17. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a spinneret into an acid, zinc-containing precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable, surface-active polymerized alkylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

18. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a spinneret into an acid precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable, surface-active polymerized alkylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited, said alkylene oxide having a polymerization degree above 30.

19. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a spinneret into an acid precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a very small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide, whereby incrustation of the spinneret during the spinning operation is substantially inhibited, said ethylene oxide having a polymerization degree above 30.

20. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a spinneret into an acid precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in an acid precipitating bath containing a small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide.

21. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a spinneret into an acid precipitating bath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments by extrusion of a viscose solution containing a small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide.

22. A viscose spin bath, for coagulating viscose filaments therein, containing a small amount of a dissolved, substantially stable, surface-active polymerized alkylene oxide.

23. A viscose spin bath, for coagulating viscose filaments therein, containing a dissolved zinc salt and a small amount of a dissolved, substantially stable, surface-active polymerized alkylene oxide.

24. A viscose spin bath, for coagulating viscose filaments therein, containing zinc sulphate and a small amount of a dissolved, substantially stable, surface-active polymerized alkylene oxide.

25. A viscose spin bath comprising water, sulphuric acid, zinc sulphate and about 0.0001 to 0.05% by weight of a dissolved, substantially stable, surface-active polymerized alkylene oxide.

26. A viscose spin bath comprising water, sulphuric acid, zinc sulphate and about 0.0001 to 0.05% by weight of a dissolved, substantially stable, surface-active polymerized ethylene oxide.

27. A viscose solution containing a small amount of a dissolved, substantially stable, surface-active polymerized alkylene oxide.

28. A viscose solution containing a small amount of a dissolved, substantially stable, surface-active polymerized alkylene oxide having a polymerization degree above 30.

29. A viscose solution containing a small amount of a dissolved, substantially stable, surface-active polymerized ethylene oxide having a polymerization degree above 30.

30. A viscose solution containing about 0.0001 to 0.05% by weight of a dissolved, substantially stable, surface-active polymerized alkylene oxide.

ARTHUR CRESSWELL.

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The following references are of record in the file of this patent:

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