

UNITED STATES PATENT OFFICE

2,294,378

SURFACE-ACTIVE INCRUSTATION INHIBITOR

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11 Claims. (Cl. 18—54)

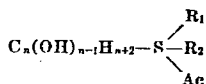
This is a continuation in part of my application Serial No. 233,826, filed October 7, 1938.

In the manufacture of cellulosic products, such as filaments, threads and the like, a viscose solution is continuously extruded through the minute orifices of a spinneret into a coagulating or setting bath. However, since the introduction of the viscose process great troubles have been and are still experienced as far as continuous spinning is concerned due to impurities, such as, for example, precipitated cellulose particles, sulphur and resin particles, zinc compounds, etc., present in either the viscose solution or the spin bath. These impurities gradually contaminate the spinnerets, clog and reduce the widths of the spinneret orifices with the result that filaments of uneven thickness are obtained. If contamination proceeds the individual filaments start to tear and occasion interruption of the spinning process.

Cation-active compounds are surface-active compounds which carry in the cation a hydrocarbon chain 8 or more carbon atoms. They are particularly advantageous because they may be used in neutral salt, alkaline and acidic aqueous solutions, and also in hard water. In contradistinction to cation-active compounds, anion-active compounds are surface-active compounds which carry in the anion a more or less extended hydrocarbon chain. They flocculate in neutral salt, alkaline and acidic aqueous solutions, and also in hard water. Common soaps, etc., are anion-active compounds, and due to their instability in acid spin baths they are unable to prevent the incrustation of spinnerets and spinneret orifices.

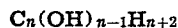
Incrustation inhibitors, in accordance with the present invention, may be prepared from sulphides derived from monosaccharides of the ketose or aldose type containing 5 or 6 carbon atoms. These sugar sulphides may be prepared from glucose, galactose, fructose, xylose, arabinose, mannose, rhamnose, ribose, talose, etc.

I have found that the contamination of spinnerets and clogging of spinneret orifices may be substantially overcome by spinning viscose solutions in the presence of cation-active monosaccharide derivatives having the general structure:

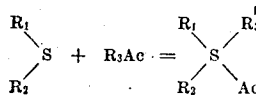


in which n is 5 or 6, R_1 is a higher aliphatic radical having 8 or more carbon atoms, R_2 represents an aliphatic or aromatic radical, and Ac

represents an innocuous anion, such as an OH group or a negative acid radical. R_2 may also represent the radical in this structure



These inhibitors may be prepared by reacting plain or mixed sulphides of monosaccharides with alkyl, aralkyl or aryl halides in conventional manner in accordance with the following equation:



Although these cation-active incrustation inhibitors may form corresponding bases in viscose solutions and salts in acid spin baths, they retain their surface-activity and, thus their property of substantially preventing incrustations on prolonged spinning. If very small amounts of these cation-active compounds are added to viscose solutions or spin baths the physical characteristics such as strength, plasticity, lustre, etc., of regenerated cellulose produced therefrom or therein, respectively, remain unaltered. Upon increasing the amount of incrustation inhibitor in a given viscose solution or spin bath, the regenerated cellulose produced therefrom or therein, respectively, may alter its physical characteristics, i. e., it may acquire a soft-lustre, become more plastic, lose part of its original strength, etc. For such reasons, the amounts to be added to viscose solutions or spin baths must be predetermined by experimentation. Additions of about 0.3 to 5.0 grams per liter of viscose solution are generally sufficient to allow continuous spinning without substantially affecting the inherent physical characteristics of regenerated cellulose threads or films. Although these cation-active incrustation inhibitors improve the spinning of any conventional viscose solution or spin bath, I have found that they are especially valuable as additions to zinc-bearing spin baths, i. e., spin baths containing a soluble zinc compound, for example, zinc sulphate, since these baths have a great tendency to incrustate metallic spinnerets. I am well aware, that cation-active glucyl amines, etc., have been heretofore prepared (vide U. S. Patents Nos. 2,016,956; 2,060,850; 2,060,851) as wetting agents. However, this prior art does not disclose the prevention of incrustations by means of cation-active compounds.

Examples of suitable incrustation inhibitors are: methyl-fructyl-octyl sulphonium chloride, ethyl-fructyl-octyl sulphonium chloride, methyl-

glucyl-octyl sulphonium bromide, methyl-glucyl-dodecyl sulphonium bromide, methyl-glucyl-dodecyl sulphonium hydroxide, ethyl-glucyl-dodecyl sulphonium sulphate, methyl-glucyl-cetyl-sulphonium bromide, phenyl-glucyl-dodecyl sulphonium bromide, benzyl-glucyl-dodecyl bromide, propyl-glucyl-octyl sulphonium iodide, methyl-galactyl-octyl sulphonium bromide, ethyl-galactyl-octyl sulphonium methyl-sulphate, etc. However, I wish to emphasize that the present invention is not limited to the compounds enumerated above, since any cation-active compound having the structure set forth above may be used provided it is sufficiently soluble and stable in viscose solutions and or acid spin baths to furnish surface-active cations therein.

Example I

About 0.1 to 5 grams of methyl-glucyl-dodecyl sulphonium bromide is added to one liter of a conventional acid spin bath. When a viscose solution is extruded therein through the fine orifices of a corrosion-resistant spinneret (for example a platinum alloy spinneret) the incrustation of the spinneret orifices is substantially inhibited.

Example II

Viscose filaments are spun in the presence of a small amount of ethyl-glucyl-dodecyl sulphonium bromide with the result that the spinneret orifices remain clean for long periods of time.

Example III

About 0.1 to 5 grams of methyl-glucyl-dodecyl sulphonium bromide is added to one liter of a conventional viscose spinning solution. When this solution is extruded into an acid spin bath to form filaments therein the spinnerets, submerged in the spin bath, remain substantially free from incrustations.

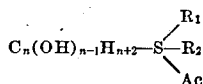
Example IV

The cation-active monosaccharide derivatives are added in small amounts to both the viscose solution and the acid spin bath in order to substantially prevent incrustation of spinneret orifices through which the viscose solution is extruded into the spin bath to form regenerated cellulose.

Modifications of my invention will readily be recognized by those skilled in the art, and I desire to include all modifications and variations coming within the scope of the appended claims.

I claim:

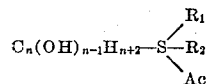
1. An acid viscose spin bath, for coagulating viscose filaments therein, containing a dissolved, substantially stable, cation-active monosaccharide derivative having the structure:



in which n is a number selected from the group consisting of 5 and 6, R_1 is a higher aliphatic radical having at least 8 carbon atoms, R_2 represents a radical selected from the group consisting of alkyl, aralkyl and aryl radicals, and Ac represents a substituent selected from the group consisting of OH and negative acid radicals, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

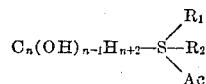
2. An acid viscose spin bath, for coagulating viscose filaments therein, containing a dissolved,

substantially stable, cation-active monosaccharide derivative having the structure:



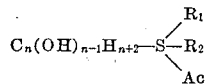
in which n is a number selected from the group consisting of 5 and 6, R_1 is a higher aliphatic radical having at least 8 carbon atoms, R_2 represents an alkyl radical, and Ac represents a substituent selected from the group consisting of OH and negative acid radicals, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

3. An acid viscose spin bath, for coagulating viscose filaments therein, containing a dissolved, substantially stable, cation-active monosaccharide derivative having the structure:



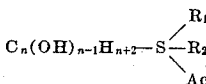
in which n is a number selected from the group consisting of 5 and 6, R_1 is a higher aliphatic radical having at least 8 carbon atoms, R_2 represents an aralkyl radical, and Ac represents a substituent selected from the group consisting of OH and negative acid radicals, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

4. An acid viscose spin bath, for coagulating viscose filaments therein, containing a dissolved, substantially stable, cation-active monosaccharide derivative having the structure:



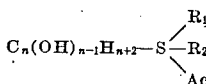
in which n is a number selected from the group consisting of 5 and 6, R_1 is a higher aliphatic radical having at least 8 carbon atoms, R_2 represents an aryl radical, and Ac represents a substituent selected from the group consisting of OH and negative acid radicals, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

5. A viscose spinning solution containing a dissolved, substantially stable, cation-active monosaccharide derivative having the structure:



in which n is a number selected from the group consisting of 5 and 6, R_1 is a higher aliphatic radical having at least 8 carbon atoms, R_2 represents a radical selected from the group consisting of alkyl, aralkyl and aryl radicals, and Ac represents a substituent selected from the group consisting of OH and negative acid radicals, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

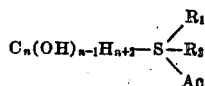
6. An acid viscose spin bath containing a water-soluble zinc compound and a dissolved, substantially stable cation-active monosaccharide derivative having the structure:



in which n is a number selected from the group consisting of 5 and 6, R_1 is a higher aliphatic radical having at least 8 carbon atoms, R_2 represents a radical selected from the group consisting

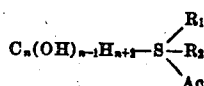
of alkyl, aralkyl and aryl radicals, and Ac represents a substituent selected from the group consisting of OH and negative acid radicals, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

7. An acid viscose spin bath containing a water soluble zinc compound and a dissolved, substantially stable cation-active monosaccharide derivative having the structure:



in which n is a number selected from the group consisting of 5 and 6, R_1 is a higher aliphatic radical having at least 8 carbon atoms, R_2 represents an alkyl radical, and Ac represents a substituent selected from the group consisting of OH and negative acid radicals, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

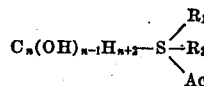
8. An acid viscose spin bath containing a water-soluble zinc compound and a dissolved, substantially stable cation-active monosaccharide derivative having the structure:



in which n is a number selected from the group consisting of 5 and 6, R_1 is a higher aliphatic radical having at least 8 carbon atoms, R_2 represents

an aralkyl radical, and Ac represents a substituent selected from the group consisting of OH and negative acid radicals, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

9. An acid viscose spin bath containing a water-soluble zinc compound and a dissolved, substantially stable cation-active monosaccharide derivative having the structure:



in which n is a number selected from the group consisting of 5 and 6, R_1 is a higher aliphatic radical having at least 8 carbon atoms, R_2 represents an aryl radical, and Ac represents a substituent selected from the group consisting of OH and negative acid radicals, whereby incrustation of the spinneret during the spinning operation is substantially inhibited.

10. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices of a 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 methyl-glucyl-dodecyl sulphonium bromide.

11. An acid viscose spin bath containing dissolved methyl-glucyl-dodecyl sulphonium bromide.

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