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COAGULATING BATH CONTAINING CATION-ACTIVE INHIBITORS

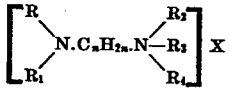
Rudolph S. Bley, Milligan College, Tenn., assignor to North American Rayon Corporation, New York, N. Y., a corporation of Delaware

No Drawing. Application October 7, 1938, Serial No. 233,824

8 Claims. (Cl. 18—54)

In the manufacture of cellulosic products, such as filaments, and the like, a viscose solution is continuously extruded through the minute orifices of a spinneret into a coagulating bath (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.

I have found that contamination of spinnerets and clogging of spinneret holes (orifices) may be substantially overcome by spinning viscose solutions in the presence of cation-active substances or acid addition products thereof having the general structure:



in which R represents an aliphatic acyl radical having 8 or more carbon atoms, R_1 represents a hydrogen atom or an acyl, alkyl, aryl or aralkyl radical, R_2, R_3 and R_4 represent alkyl and/or aryl and/or aralkyl radicals, n is a whole number, and X represents an innocuous anion.

Cation-active compounds are surface-active compounds which carry in the cation a hydrocarbon chain having 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, sulphonated oils, etc., are anion-active compounds, and thus they are unable to prevent the incrustation of spinnerets and spinneret orifices.

Incrustation inhibitors, in accordance with the present invention, may be prepared by causing high-molecular, mono-acylated or unsymmetrical diacylated alkylenediamines with alkylating, arylating or aralkylating agents, such as dimethyl sulphate, methyl iodide, methyl bromide, methyl chloride, ethyl iodide, ethyl bromide, propyl bromide, benzyl chloride, benzyl bromide, phenyl bromide, p-toluene-ethyl-sulphonate, etc. The acetylated or acylated alkylenediamines may be prepared in accordance with processes set forth in U. S. Patent No. 1,534,525.

Although these cation-active incrustration 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 incrustrations 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 and/or spin baths must be predetermined by experimentation. Additions of about 0.1 to 5.0 grams per liter of viscose solution or spin bath are generally sufficient to allow continuous spinning without substantially affecting the inherent physical characteristics of regenerated cellulose threads, films, etc. 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, these baths having poor spinning qualities. I am well aware that cation-active alkylene diamines have, heretofore, been prepared as wetting agents. However, the prior art does not disclose the use of these derivatives as incrustation inhibitors, the object of the present invention.

Example I

About 380 parts of oleyl-diethylethylenediamine are reacted with about 126 parts of dimethyl-sulphate. About 0.3 to 2.0 grams of the oily mono - oleyl - diethyl - methylethylene - diammonium-methyl-sulphate are added to about one liter of a viscose solution of conventional cellulose content and maturity. This solution is then extruded through a spinneret into a conventional spin bath, such as, for example, a glucose bath, a magnesium-zinc bath, etc. The spinnerets and

their orifices remain clean on prolonged spinning. Other cation-active alkylenediamine derivatives having the general structure set forth above may be used with equal success.

Example II

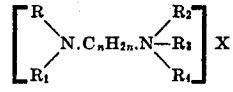
About 382 parts of stearyl-diethylethylenediamine are melted and reacted with about 126 parts of dimethyl sulphate. The soapy mass is probably mono-stearyl-diethyl-methylethylene-di-ammonium-methyl-sulphate.

A conventional viscose solution is extruded through fine orifices of precious metal spinnerets into an acid spin bath, containing for example, sulphuric acid, sodium sulphate, ammonium sulphate and zinc sulphate and about 0.3 to 5.0 grams per liter of mono-stearyl-diethyl-methyl-ethylene-diammonium-methyl-sulphate. Spinning difficulties and irregularities are substantially overcome. Without the addition of an incrustation inhibitor spinning is interrupted after a very short period of time. This inhibitor may be replaced by similar derivatives obtained from lauryl-diethylenediamine, octanoyl-diethylenediamine, lauryl-dipropylenediamine, octanoyl-dipropylenediamine, etc.

Example III

380 parts of oleyl-diethylethylenediamine are reacted with about 142 parts of benzyl chloride. The resulting oily product, probably mono-oleyl-diethyl-benzylethylene-diammonium-chloride, is added to both the viscose solution and the spin bath. The formation of zinc rings and sulphur craters is substantially overcome.

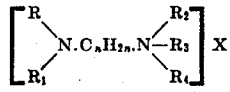
Although these examples will serve to illustrate the present invention, I do not wish to be limited to the incrustation inhibitors and concentrations thereof recited therein, since I may make use of any cation-active alkylene derivative and acid addition products thereof having the structure:



in which R represents an aliphatic acyl radical having 8 or more carbon atoms, R_1 represents a hydrogen atom or an acyl, alkyl, aryl or aralkyl radical, R_2, R_3 and R_4 represent alkyl and/or aryl and/or aralkyl radicals, n is a whole number, and X represents an innocuous anion, provided it is sufficiently soluble and stable in spin baths and/or viscose solutions to furnish surface-active cations therein. Modifications of my invention will readily be recognized by those skilled in the art, and I desire to include all such modifications coming within the scope of the appended claims.

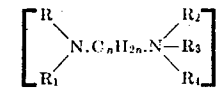
I claim:

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



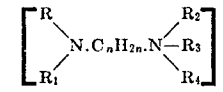
in which R represents an aliphatic acyl radical having 8 or more carbon atoms, R_1 represents a substituent selected from the group consisting of hydrogen, acyl radicals, alkyl radicals, aryl radicals and aralkyl radicals, R_2, R_3 and R_4 represent a radical selected from the group consisting of alkyl radicals, aryl radicals and aralkyl radicals, n is a whole number and X represents an innocuous anion.

2. An acid viscose spin bath, for coagulating viscose filaments therein, containing a dissolved zinc salt and a dissolved, substantially stable, cation-active alkylene derivative having the structure:



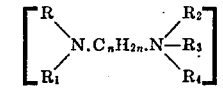
in which R represents an aliphatic acyl radical having 8 or more carbon atoms, R_1 represents a substituent selected from the group consisting of hydrogen, acyl radicals, alkyl radicals, aryl radicals and aralkyl radicals, R_2, R_3 and R_4 represent alkyl radicals, n is a whole number and X represents an innocous anion.

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



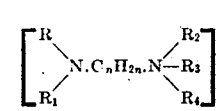
in which R represents an aliphatic acyl radical having 8 or more carbon atoms, R_1 represents a substituent selected from the group consisting of hydrogen, acyl radicals, alkyl radicals, aryl radicals and aralkyl radicals, R_2, R_3 and R_4 represent aryl radicals, n is a whole number and X represents an innocuous anion.

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



in which R represents an aliphatic acyl radical having 8 or more carbon atoms, R_1 represents a substituent selected from the group consisting of hydrogen, acyl radicals, alkyl radicals, aryl radicals and aralkyl radicals, R_2, R_3 and R_4 represent aralkyl radicals, n is a whole number and X represents an innocuous anion.

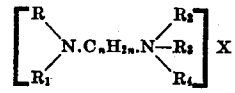
5. An acid viscose spin bath for coagulating viscose filaments therein comprising sulphuric acid, zinc sulphate, water and about 0.1 to 5.0 grams per liter of a dissolved, substantially stable, cation-active alkylene derivative having the structure:



in which R represents an aliphatic acyl radical having 8 or more carbon atoms, R_1 represents a substituent selected from the group consisting of hydrogen, acyl radicals, alkyl radicals, aryl radicals and aralkyl radicals, R_2, R_3 and R_4 represent a radical selected from the group consisting of alkyl radicals, aryl radicals and aralkyl radicals, n is a whole number and X represents an innocuous anion.

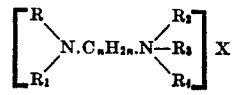
6. An acid viscose spin bath for coagulating viscose filaments therein comprising sulphuric acid, zinc sulphate, water and about 0.1 to 5.0 grams per liter of a dissolved, substantially stable,

cation-active alkylene derivative having the structure:



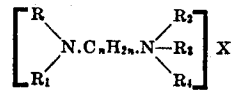
in which R represents an aliphatic acyl radical having 8 or more carbon atoms, R_1 represents a substituent selected from the group consisting of hydrogen, acyl radicals, alkyl radicals, aryl radicals and aralkyl radicals, R_2, R_3 and R_4 represent alkyl radicals, n is a whole number and X represents an innocuous anion.

7. An acid viscose spin bath for coagulating viscose filaments therein comprising sulphuric acid, zinc sulphate, water and about 0.1 to 5.0 grams per liter of a dissolved, substantially stable, cation-active alkylene derivative having the structure:



in which R represents an aliphatic acyl radical having 8 or more carbon atoms, R_1 represents a substituent selected from the group consisting of hydrogen, acyl radicals, alkyl radicals, aryl radicals and aralkyl radicals, R_2, R_3 and R_4 represent aryl radicals, n is a whole number and X represents an innocuous anion.

8. An acid viscose spin bath for coagulating viscose filaments therein comprising sulphuric acid, zinc sulphate, water and about 0.1 to 5.0 grams per liter of a dissolved, substantially stable, cation-active alkylene derivative having the structure:



in which R represents an aliphatic acyl radical having 8 or more carbon atoms, R_1 represents a substituent selected from the group consisting of hydrogen, acyl radicals, alkyl radicals, aryl radicals and aralkyl radicals, R_2, R_3 and R_4 represent aralkyl radicals, n is a whole number and X represents an innocuous anion.

RUDOLPH S. BLEY.