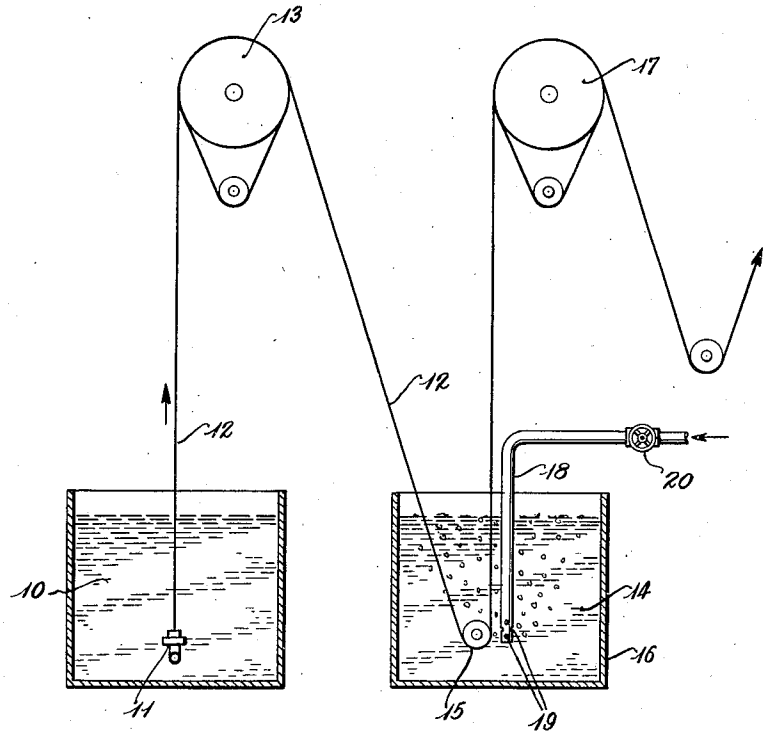
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INHIBITIONS OF INCRUSTATIONS

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INHIBITION OF INCRUSTATIONS

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This invention relates to the manufacture of rayon, and more particularly to the production of viscose rayon by the two-bath method. The advantages of the so-called two-bath method of producing viscose rayon are widely known and accepted in the art, particularly in connection with the manufacture of high tenacity yarn for the production of reinforcements for cord tires and the like. However, it has been found in practice that the normal two-bath spinning process involves the formation of crusts upon surfaces of the elements with which the second bath contacts. The formation of such crusts upon the thread guiding surfaces constitutes a hazard to the newly formed yarns or threads and the crusts create additional problems when the operation is continued over long periods without interruption for cleaning the equipment.

In general, the two-bath system of manufacturing viscose rayon yarn involves the extrusion of a viscose solution through a spinneret into a coagulating bath wherein the freshly formed viscose filaments are partially regenerated, the filaments being then directed into a second hot bath, after which they are collected in a pot or on a bobbin or by some other suitable collection mechanism. During the passage of the bundle of filaments through the second bath, it is given a high stretch for the purpose of imparting to the yarn or thread an increased tensile strength.

The initial bath, that is, the bath into which the viscose solution is extruded, normally contains a substantial proportion of zinc sulphate. The zinc sulphate is added to the first bath for the purpose of retarding regeneration of the thread. In the preparation of the second bath, zinc sulphate is not added thereto but it has been found in practice that the yarn or thread as it passes from the first bath to the second bath carries over into the second bath components of the first bath which include a portion of the zinc sulphate. During the spinning operation, hydrogen sulphide is evolved in both the first and second baths. Since zinc sulphate is present in the second bath as a result of the carry over from the first bath, zinc sulphide is formed therein as a result of the reaction of hydrogen sulphide with zinc sulphate. The second bath contains but a very small amount of sulphuric acid, usually of the order of about 1.0%, while the first bath normally contains a sulphuric acid concentration of about 9.5%. In the first bath having the high concentration of sulphuric acid the problem of crust formation is not present but in the second bath of low acid concentration the solubility product of zinc sulphide is exceeded and crust formations are produced. This problem encountered in the use of the two-bath method has been particularly pronounced when zinc sulphate is present in the first bath in an amount in excess of 1.0%.

The hard crusts formed in the second bath occur on the walls of the bath trough and on the other equipment with which the second bath contacts. This equipment includes the lead slides and the plastic thread guiding rollers which are submerged in the second bath. Additionally, the effluent pipe lines are also affected by the crust formation, and in fact when an operation has been continued over a long period of time, the crust gradually builds up in the pipe lines to a degree where the lines are almost completely clogged.

In an operation in which the second bath is recirculated even the supply lines tend to become partially clogged. It is clear, therefore, that in such an operation the formation of the crusts or deposits requires frequent cleaning particularly of the guiding rollers in order to prevent physical damage to the thread or yarn.

Of course, the formation of the crust can be prevented when the second bath is replenished with fresh liquid in sufficient quantities to prevent the concentration of reactants from exceeding the solubility product of zinc sulphide. However, such practice is not commercially feasible due to the waste of materials and the resultant expense.

The present invention has as an object the avoidance of the formation of crusts of the type above referred to in the second bath of a two-bath process for the production of viscose rayon. The invention contemplates the avoidance of the formation of such crusts in the said second bath even over long periods of operation of processes in which the zinc sulphate content of the first bath exceeds 1.0% concentration.

It is an object of the present invention to provide a process for producing viscose rayon by the two-bath procedure in which the zinc content is present in the second bath in an amount such as to normaly affect the formation of objectionable crusts, but in which the bath may be recirculated without incurring the clogging of the supply and exit pipe lines, and the occurrence of crusts upon the walls of the bath container, as well as upon the thread guiding elements, is avoided.

Other objects and advantages will be apparent from the following detailed description when

considered in connection with the accompanying drawing, wherein the single figure is a diagrammatic illustration of a two-bath viscose spinning system with the present invention illustrated in connection therewith.

Referring to the drawing, the first bath designated by numeral 10 has arranged therein a spinneret 11. A viscose solution is pumped from the usual source (not illustrated) and is extruded from spinneret 11 in the form of a bundle of filaments or yarn 12. This yarn or thread 12 is then directed to a driven godet 13 in the usual manner. Coagulating bath 10 contains the normal bath constituents, including zinc sulphate within the range of from about 1.0% to 5.0% and preferably of the order of about 2.8%.

Thread 12, after leaving godet 13, is passed into bath 14 and around submerged roller 15, the bath being contained in trough 16. From roller 15 thread 12 is fed to godet 17, after which it is directed to a suitable collecting mechanism.

Bath 14 is preferably of low acid concentration and it is maintained at a temperature of between 70° C. and 90° C., preferably about 80° C. The acid is preferably sulphuric acid and maintained at a concentration in a range of 0.3 to 1.0% based on the weight of the bath. As thread 12 is propelled through bath 14, it is simultaneously stretched, and if desired is then subjected continuously to one or all of the normal after-treating operations before it is collected as a package. However, if desired, the thread may be collected and after-treated in package form.

Pipe 18 having a series of perforations 19 adjacent its lower end at a level below the surface of bath 14 is connected to a source of air or other gas under sufficient pressure to cause the same to be forced into bath 14 and to bubble therethrough. If desired, a series of pipes may be employed for the introduction of air into the bath. The amount of air admitted to bath 14 may be controlled by valve 20, or in any other convenient manner.

For the purpose of filtering bath 14 and for replenishing the same, exit and supply lines (not shown) are connected in the wall of trough 16.

In practice, during the continuous spinning operation any suitable gas such as air or nitrogen is supplied under pressure to the bath 14 and the bubbling of the gas therethrough causes the bath to be continuously agitated. Hydrogen sulphide is thereby continuously removed from the bath. Since hydrogen sulphide is one of the reacting components, this removal of the same from the bath inhibits the formation of incrustations on the surfaces of the equipment, and as the bath is recirculated it is only necessary to replenish the same to an extent sufficient to maintain the desired acid concentration. Furthermore, the use of the same bath without the necessity of replenishing the same with large quantities of fresh liquid effects a great saving from the standpoint of heat loss.

By way of example, in practicing one specific process in accordance with the present invention, a normal viscose solution was extruded through a spinneret in a conventional manner into a spinning bath of the following composition, the percentages given being calculated on the weight of the bath:

	Percent
Sulphuric acid	9.5
Sodium sulphate	17.3
Magnesium sulphate	4.9
Zinc sulphate	2.8
Water	65.5

The process involved the production of 480 filament 1100 denier yarn. The bundle of filaments was withdrawn without slippage from the bath. Regeneration of the cellulose xanthate in the yarn to cellulose was retarded by the employment of the particular spinning bath, and the partially regenerated filaments, after passing from the drawoff godet, were immersed in a hot, dilute acid bath containing sulphuric acid in a range of 0.8 to 1.0%.

Throughout the run, which extended over a period of 21 days, air was blown into the second bath, the volume of which bath was 13 liters. Through a distribution pipe in the manner illustrated in the accompanying drawing, air was delivered to the second bath. Said bath was supplied and replenished only to the extent necessary to dilute the acid carried over by the thread in order to maintain the concentration at about 1.0%, based on the weight of the bath. The air was continuously dispersed throughout the bath at a rate of 1.25 liters per spinning position per minute. During the entire run the second bath was maintained at a temperature of about 80° C. and a stretch of about 75% was given to the yarn in its passage through said bath.

At the end of the run the thread guiding rollers were found to be clean and only a thin soft film was found on the walls of the trough and the roller slides, as distinguished from the hard, thick, heavy crust which had been found in similar runs where air was not bubbled through the second bath. An analysis of the second bath of this run showed a hydrogen sulphide content of 24 milligrams per liter as compared with 60 milligrams per liter in a similar run in which the present invention was not utilized.

Whereas the invention has been described in its preferred form, various modifications may be made without departing from the spirit and scope thereof and it is to be understood that this invention is only limited to the extent of the appended claims.

What is claimed is:

1. A process of manufacturing rayon, comprising the passing of incompletely regenerated viscose through a bath of low acid concentration in which hydrogen sulphide is evolved, zinc sulphate being present in said bath in an amount sufficient to form zinc sulphide in an amount exceeding the solubility product of zinc sulphide in the bath, removing hydrogen sulphide by agitating the bath with a current of air, whereby the hydrogen sulphide content of the bath is so reduced that the reaction of the zinc sulphate with the hydrogen sulphide remaining does not form an amount of zinc sulphide exceeding its solubility product in the bath.

2. A process of manufacturing rayon, comprising the passing of incompletely regenerated viscose through a bath of an acid concentration of about 1.0% based on the weight of the bath, in which hydrogen sulphide is evolved, zinc sulphate being present in said bath in an amount sufficient to form zinc sulphide in an amount exceeding the solubility product of zinc sulphide in the bath, removing hydrogen sulphide by agitating the bath with a current of air, whereby the hydrogen sulphide content of the bath is so reduced that the reaction of the zinc sulphate with the hydrogen sulphide remaining does not form an amount of zinc sulphide exceeding its solubility product in the bath.

3. A process of manufacturing rayon, comprising the passing of incompletely regenerated

viscose through a bath of low acid concentration in which hydrogen sulphide is evolved, zinc sulphate being present in said bath in an amount sufficient to form zinc sulphide in an amount exceeding the solubility product of zinc sulphide in the bath, removing hydrogen sulphide by agitating the bath with a current of air, whereby the hydrogen sulphide content of the bath is maintained at about 24 milligrams per liter of the said bath so that the reaction of the zinc sulphate with the hydrogen sulphide remaining does not form an amount of zinc sulphide exceeding its solubility product in the bath.

4. A process of manufacturing rayon, comprising the passing of incompletely regenerated viscose through a bath of an acid concentration of about 1.0% based on the weight of the bath, in which hydrogen sulphide is evolved, zinc sulphate being present in said bath in an amount sufficient to form zinc sulphide in an amount exceeding the solubility product of zinc sulphide in the bath, removing hydrogen sulphide by agitating the bath with a current of air, whereby the hydrogen sulphide content of the bath is maintained at about 24 milligrams per liter of the said bath so that the reaction of the zinc sulphate with the hydrogen sulphide remaining does not form an amount of zinc sulphide exceeding its solubility product in the bath.

5. A process of manufacturing rayon, comprising the passing of incompletely regenerated viscose through a bath of low acid concentration in which hydrogen sulphide is evolved, maintaining the said bath within a temperature range of about 70° C. to 90° C., zinc sulphate being present in said bath in an amount sufficient to form zinc sulphide in an amount exceeding the solubility product of zinc sulphide in the bath, removing hydrogen sulphide by agitating the bath with a current of air, whereby the hydrogen sulphide content of the bath is so reduced that the reaction of the zinc sulphate with the hydrogen sulphide remaining does not form an amount of zinc sulphide exceeding its solubility product in the bath.

6. A process for the manufacture of viscose rayon comprising extruding a viscose solution into an acid spin bath having an acid concentration of the order of about 9.5% and a zinc sulphate content of between 1.0% and 5.0% withdrawing the filaments thus formed as a thread from the said bath and passing the same through a second regenerating bath maintained at a temperature between about 70° C. and 90° C., said second bath having an acid range of about 0.3% to 1.0% sulphuric acid and in which hydrogen sulphide is evolved, the hydrogen sulphide evolved being in such amount as to normally cause a reaction with the zinc sulphate carried over to the second bath from the spin bath to form zinc sulphide in excess of the solubility product in the second bath, and forcing a current of air through the second bath to remove hydrogen sulphide evolved so that said second bath only retains a hydrogen sulphide content below that necessary to react with the zinc sulphate to exceed the solubility product of zinc sulphide in the second bath.

7. A process for the manufacture of viscose rayon comprising extruding a viscose solution into an acid spin bath having an acid concentration of the order of about 9.5% and a zinc sulphate content of about 2.8%, withdrawing the filaments thus formed as a thread from the said bath and passing the same through a second regenerating bath maintained at a temperature between 70° C. and 90° C., said second bath having an acid concentration of about 1.0% sulphuric acid and in which hydrogen sulphide is evolved, the hydrogen sulphide evolved being in such amount as to normally cause a reaction with the zinc sulphate carried over to the second bath from the spin bath to form zinc sulphide in excess of the solubility product in the second bath, forcing a current of air through the second bath to remove hydrogen sulphide evolved so that said second bath only retains a hydrogen sulphide content below that necessary to react with the zinc sulphate to exceed the solubility product of zinc sulphide in the second bath.

8. A process for the manufacture of viscose rayon comprising extruding a viscose solution into an acid spin bath having an acid concentration of the order of about 9.5% and a zinc sulphate content of between 1.0% and 5.0%, withdrawing the filaments as a thread from the said bath and passing the same through a second regenerating bath maintained at a temperature of about 80° C., said second bath having an acid concentration of about 1.0% sulphuric acid and in which hydrogen sulphide is evolved, the hydrogen sulphide evolved being in such amount as to normally cause a reaction with the zinc sulphate carried over to the second bath from the spin bath to form zinc sulphide in excess of the solubility product in the second bath, and forcing a current of air through the second bath to remove hydrogen sulphide evolved so that said second bath only retains a hydrogen sulphide content of about 24 milligrams per liter, whereby the hydrogen sulphide content retained does not react with the zinc sulphate to exceed the solubility product of zinc sulphide in the second bath.

9. A process for the manufacture of viscose rayon comprising forming filaments by extruding a viscose solution into an acid spin bath having an acid concentration of the order of about 9.0% and a zinc sulphate content exceeding about 1.0%, withdrawing the filaments as a thread from the said bath and passing the same through a second regenerating bath maintained at a temperature between about 70° C. and 90° C., said second bath having a low acid concentration and evolving hydrogen sulphide, the hydrogen sulphide evolved being in such amount as to normally cause a reaction with the zinc sulphate carried over to the second bath from the coagulating bath to form zinc sulphide in excess of the solubility product in the second bath, and forcing a current of air through the second bath to remove hydrogen sulphide as evolved so that said second bath only retains a hydrogen sulphide content of about 24 milligrams per liter, whereby the hydrogen sulphide content retained does not react with the zinc sulphate to exceed the solubility product of zinc sulphide in the second bath.

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