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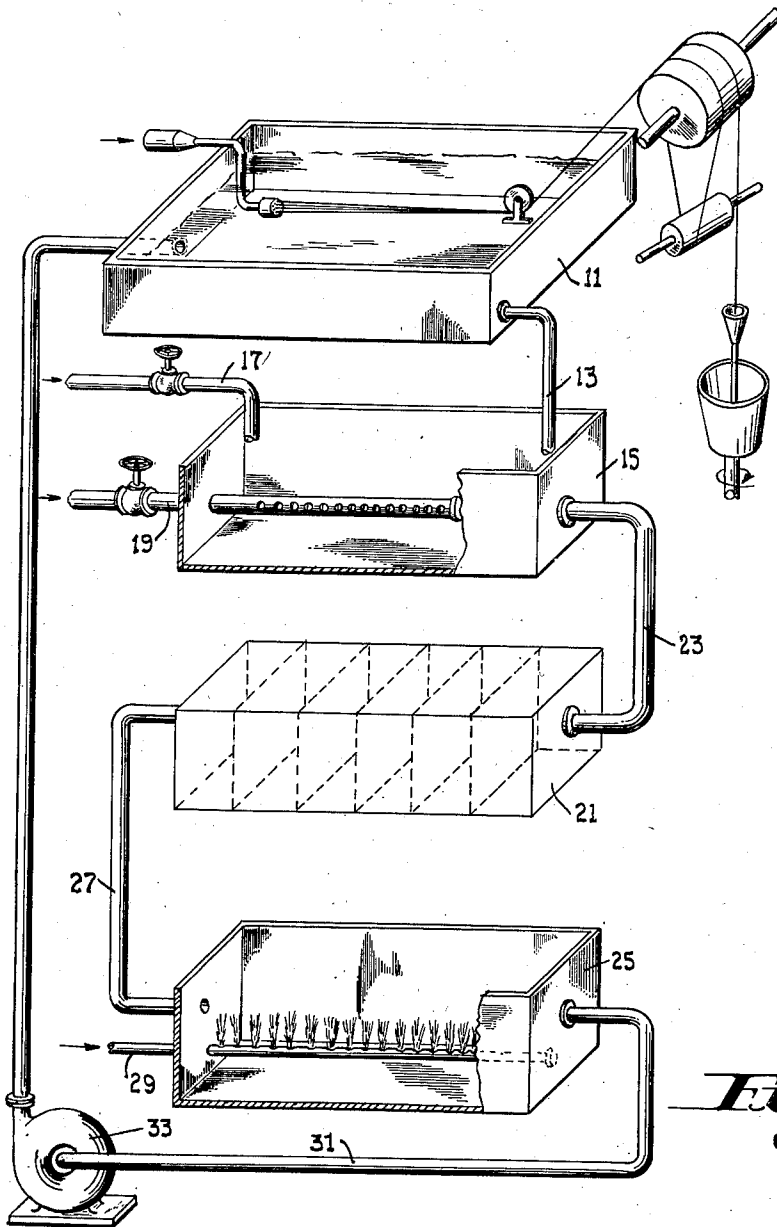


Fig. 1

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YARN PRODUCTION

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This invention relates to the manufacture of regenerated cellulose filaments by the viscose process and, more specifically, it relates to an improved process for spinning such filaments in continuous fashion without the interruptions and delays occasioned by the deposition of foreign materials at the spinneret orifices. It relates more particularly to a process for spinning regenerated cellulose filaments from viscose while materially diminishing the formation and deposition of foreign materials at the spinneret orifices during the spinning operation.

In the manufacture of artificial filaments by the viscose process, it is customary to extrude a viscose solution through spinneret orifices into an aqueous acid coagulating and regenerating bath contained in a lead trough or tank, whence the formed filaments are withdrawn and ultimately collected in the form of a suitable package.

The formation and deposition of foreign materials on the face of the spinneret, and even within the perforations of the spinneret which constitute the spinning orifices, has long been a troublesome factor in the spinning of artificial filaments by the viscose process. These deposits, sometimes referred to as "spinneret craters," interfere with the continuity of the spinning operations in numerous ways. For example, deposits within the spinneret orifices diminish the size thereof, causing small, weak filaments to form, which frequently break during subsequent travel through the regenerating bath or during other after-treatments. Sometimes also the spinneret orifices become so completely obstructed that little or no viscose can pass through, resulting in an off-standard product. The rapidity with which these deposits collect and the frequency of spinneret changes required thereby constitute serious obstacles in the way of uniform, economical spinning operations, not to mention the uniformity of the product.

Heretofore, the presence of insoluble material in the regenerating bath has been suspected as a cause for spinneret obstruction and resort taken to periodic filtration of the bath to clarify the same by removal of insoluble precipitated material.

Heretofore, it has also been proposed to improve the spinning performance by adding various substances to the viscose or to the regenerating bath in an attempt to prevent adhesion of these foreign materials to the spinneret face or orifices. These proposals have met with some success but they have the serious disadvantage

of further complicating an already complicated chemical composition, either as viscose or as regenerating bath. Furthermore, these substances are often prohibitively expensive to use.

It has now been observed that mere filtration of insoluble materials from the bath has little or no retarding effect on the deposition of foreign materials at the spinneret orifices. In other words, it appears that the difficulty arises, not so much from the deposition of materials already suspended in the regenerating bath, as from the deposition of insoluble materials, as they are formed, in the reaction zone at the face of the spinneret. Consequently, one might filter the bath to obtain a clear colorless solution for many years and still suffer from the deposition of foreign materials at the spinneret orifices without realizing that the formation of insoluble salts in the reaction zone at the spinneret face is the critical factor. Furthermore, it has now been discovered that the deposition of foreign material on the face of the spinneret can be substantially diminished if the heavy metal ion content of the bath is reduced and maintained sufficiently low to substantially eliminate the formation of an insoluble heavy metal salt in the reaction zone at the face of the spinneret. In the conventional processes for the production of regenerated cellulose from viscose the presence of lead and sulfide ions in sufficient quantity to form lead sulfide on the spinneret has now been found to be the cause of particularly objectionable deposits of foreign material on the spinneret. It is probable that materials already suspended in the bath will tend to adhere to the spinneret face more readily when conditions are such that insoluble salts are formed in the reaction zone adjacent the spinneret face, since they may even be occluded in the deposition of the freshly formed insoluble material.

That lead sulfide should be the principal offender in the viscose process as described is surprising. As is well known to those skilled in the art, the solubility product of lead sulfide is affected by the hydrogen ion concentration in an acid system, such as a viscose process spinning bath. Likewise, the presence of other electrolytes or changes in temperature will cause it (the solubility product) to vary. It is well known that lead sulfide will not precipitate readily in the presence of high concentrations of hydrogen ions. Judged by solubility characteristics alone, lead sulfide would not be expected to precipitate out of a viscose process spinning bath. However, and this is a significant point which apparently has

been overlooked in the prior art, at the reaction zone in the vicinity of the spinneret face, because of the interaction of the sodium hydroxide of the viscose and the sulfuric acid of the spinning bath, the hydrogen ion concentration will be low enough to permit some precipitation of lead sulfide if the concentrations of the lead and sulfide ions are sufficiently high so that the solubility product under those conditions is exceeded whatever it may be in absolute value. Once formed, the lead sulfide is difficult to redissolve and may be removed to considerable extent by later filtration.

In the commercially operated viscose rayon processes, the commonly employed regenerating bath comprises an aqueous solution of sulfuric acid, sodium sulfate, zinc sulfate and glucose. The zinc sulfate and/or glucose are sometimes omitted but their presence is preferred for good spinning performance. The presence of glucose permits reduction in the amount of sodium sulfate required. The zinc sulfate, which is sometimes supplemented in its action by the addition of nickel, cobalt or ferrous iron sulfates, makes possible the production of filaments of higher tenacity.

In addition to these desirable and normally controlled components of the regenerating bath, there are also present other materials which derive from the impurities in the raw materials used in making up the bath, from the vessels and mechanical equipment which contain or handle the bath in use, and from impurities in the viscose, which materials accumulate in the bath because of the customary practice of re-using the bath in an economical spinning operation. As above indicated, it has now been found that the most objectionable among the normally uncontrolled components of the regenerating bath are lead- and sulfur-containing compounds.

The viscose employed usually comprises principally an aqueous sodium hydroxide dispersion of sodium cellulose xanthate but because of the unstable character of sodium cellulose xanthate, it contains varying amounts of sodium sulfide, thiocarbonates, etc.

When the viscose is brought into contact with the regenerating bath, there occur, of course, a number of chemical reactions which result in the formation of hydrogen sulfide, carbon disulfide, sulfur, thio acids, etc., in addition to the regeneration of the cellulose. Moreover, when the bath contains lead, as discussed above, the further formation of insoluble lead sulfide is indicated. Thus, it becomes evident that those components of the bath and viscose which, heretofore, have been left uncontrolled, offer ample opportunity for the formation of insoluble materials in the reaction zone at the face of the spinneret, which materials, formed in that zone, may deposit on the spinneret face or in the spinneret orifices and, in some instances, colloidal or other suspended particles in the bath may be deposited simultaneously therewith so that comparatively rapid obstruction of the spinning orifices results.

The equipment used for handling the bath is principally lead or lead-lined equipment. The amount of lead in the bath depends then on the lead content of the raw materials, the type of lead (i. e., hard lead or chemically pure lead) of the equipment, particularly when lead heating coils are used, the ratio of the volume of the bath to the area of exposed lead, the type and degree of filtration of the bath and even on the amount of yarn being spun. The sulfuric acid

employed frequently contains lead sulfate and may, therefore, be the source of both lead and sulfate ions in the bath.

It is, therefore, the object of this invention to substantially diminish the formation and deposition of foreign materials at the spinneret face in the spinning of regenerated cellulose by the viscose process.

Other objects of the invention will appear hereinafter.

The objects of the invention may be accomplished, in general, by reducing the heavy metal ion content, and preferably also the sulfide content, of the coagulating bath in a viscose rayon process, and maintaining the heavy metal ion content, and preferably also the sulfide content, of said bath sufficiently low to substantially eliminate the formation of an insoluble heavy metal salt, particularly an insoluble metal sulfide, in the reaction zone at the face of the spinneret.

The accompanying illustration is a diagrammatic perspective view showing one embodiment of apparatus suitable for use in carrying out the process of the invention.

Referring to the drawing, reference numeral 11 designates a bath container for a coagulating and regenerating bath of a viscose rayon spinning process. The spinning bath 11 is connected by means of conduit 13 to a receiving tank 15. The bath in the receiving tank 15 may be replenished with fresh bath constituents through conduit 17. If desired, the bath in receiving tank 15 may be saturated with hydrogen sulfide by means of perforated conduit 19. The receiving tank 15 is connected to a filter 21 by means of conduit 23. The filter 21 is connected to an aeration tank 25 by conduit 27. Air may be passed into the bath in the aeration tank 25 through perforated conduit 29. From the aeration tank 25, the bath liquid can be returned to the coagulating bath tank 11 through conduit 31 and pump 33.

The following examples are given to illustrate preferred methods for practicing the present invention, it being understood, however, that the present invention is not limited to the detailed methods set forth therein.

Example I

A suitable viscose solution, containing 7% cellulose and 6% sodium hydroxide and containing a delustering agent, if desired, is spun in the customary fashion to yield a 100-denier, 60-filament yarn using a platinum-gold alloy spinneret having orifice diameters of 0.0035 of an inch and a coagulating and regenerating bath containing 9% sulfuric acid, 19% sodium sulfate, 0.7% zinc sulfate and 4% glucose. The bath, before recirculation to the spinning positions, is led to a receiving tank where it is customary to add the usual replenishing materials, such as acid, glucose, or the like, part of which may be fresh materials and part the liquors from evaporation and crystallization treatments employed to remove excess soluble salts, such as sodium sulfate, in accordance with the well-known procedure of the art. The bath is then filtered at a suitable rate through an anthracite filter prepared as described in detail below. From the filter the bath is led to a large capacity aeration tank, for example, a tank having a capacity of 300 gallons, where it is aerated as hereinafter described using an air flow of about 1 cubic foot per minute for every 5 to 6 gallons per minute flow of bath through the tank, the air being forced in under a pressure of about 20

pounds per square inch. While in the aeration tank, the bath is also heated sufficiently to raise its temperature to about 45° C. and then it is led to the spinning machine. As the bath passes to the first spinneret of a spinning machine having 50 spinnerets, it has a lead content of about 3 parts per million and a sulfide ion content of not more than 10 parts per million. The sulfide ion content is measured by the following iodometric method:

Ten milliliters of 0.1N iodine solution are pipetted into a 300-milliliter bottle provided with a ground glass stopper. To this are added 100 milliliters of bath and the mixture is allowed to stand for exactly 10 minutes in the stoppered bottle. The mixture is then quickly titrated with 0.1N sodium thiosulfate solution until the color changes to a light brown. At this point 5 milliliters of starch indicator are added and the titration is completed. Care must be taken to avoid over-running the end point because the sample itself is not always colorless. As a blank titration, 10 milliliters of distilled water and 5 milliliters of 36N H₂SO₄ are added to the 10 milliliters of 0.1N iodine solution in a 250-milliliter flask and the whole is titrated with 0.1N sodium thiosulfate. The consumed iodine in the test titration, corrected for the blank titration, measures the reducing power of the bath in milliliters of 0.1N iodine per 100 milliliters of bath. Calculating the reducing power as a function of sulfide ion concentration, 1 milliliter of 0.1N iodine solution per 100 milliliters of bath is equivalent to 16 parts per million of sulfide ion divided by the density of the bath.

Example II

A delustered yarn of 100 denier, 100 filaments is produced by spinning a viscose comprising 7% cellulose, 6% sodium hydroxide, about 0.1% titanium dioxide and 0.3% white mineral oil into a spinning bath having the composition of 11% sulfuric acid, 21% sodium sulfate, 4% glucose and 0.7% zinc sulfate at a draw-off speed of 3,000 inches per minute. The bath recirculated to the spinning machine is reconditioned in the manner described in Example I and as it passes to the first spinneret, it has a lead content of about 5 parts per million and a sulfide content of about 2 parts per million. Yarn is spun for a long period of time without encountering spinning difficulties caused by deposition of solid materials at the face of the spinneret. An even denier yarn is obtained and substantially no broken filaments were encountered.

Example III

A semi-delustered yarn (100-denier, 100-filament) is spun from viscose prepared from highly purified wood pulp (7% cellulose, 6% sodium hydroxide) and containing dibeta-naphthol ether of ethylene glycol as a delusterant (about 0.1%) using a spinning bath similar to that of Example II except that it contains 23% sodium sulfate. A draw-off speed of 3300 inches per minute is employed. The bath, reconditioned as in Example I, approaches the first spinneret with a lead content of about 3.5 parts per million and a sulfide content of about 15 parts per million. An even denier yarn is spun with substantially no broken filaments for a very considerable period of time without encountering spinning difficulties caused by deposition of solid materials at the spinneret face.

Example IV

A delustered yarn is spun as described in Example III using a draw-off speed of 4000 inches per minute. The recirculated bath, reconditioned as described in Example I, approaches the first spinneret with a lead content of about 3 parts per million and a sulfide content of about 12 parts per million. Over a comparatively long spinning period, no difficulties are encountered caused by deposition of solid materials at the spinneret face.

In all of the above examples a conventional lead spinning trough, lead heating coils and other usual equipment are employed. The spinnerets are operated over a period of 24 to 72 hours. Experience with similar spinning conditions but employing a bath containing lead and sulfide in quantities normally contained in such a bath necessitates cleaning and changing of spinnerets about once every eight hours.

The filtration of the bath is carried out with the use of a carefully prepared filter and under such conditions as to remove as much suspended material as possible, including insoluble lead sulfide, lead sulfate, and sulfur. Filtration of the bath is critical in the sense that it should be carefully done to insure substantially complete removal of the insoluble materials. Any type of filter may be employed if it is carefully prepared and contains sufficient filtering material for the purpose specified. For example, satisfactory filters may be prepared with carefully packed excelsior, or wood shavings, sand, activated carbon, or the like. In practice, excellent results have been obtained by the use of a filter made up of selected graded sizes of anthracite. Settling tanks, centrifugal force, or any other means of clarification may be employed, where practical, to effect the same result. The turbidity of the filtrate should be as low as possible and should preferably not exceed 25 parts per million as determined by the methods described in "Standard Methods of Water Analysis" (8th ed., 1936, American Public Health Association), if the maximum benefits of the invention are to be realized.

Particularly good filtration is obtained, as mentioned above, by the use of an anthracite filter. Such a filter unit may advantageously consist of a closed tank, about 8 feet in diameter, essentially cylindrical but having suitably concave ends which serve as the top and bottom of the tank. The cylindrical middle portion is about 6 feet, 7 inches in altitude. Inside the tank and supported on a suitable grid or screen the filter bed is prepared in a manner similar to a conventional sand or gravel filter. Several layers of anthracite, preferably treated with dilute sulfuric acid to remove iron and thoroughly washed, are arranged as follows from bottom to top:

- 6 inch layer nut coal
- 4 inch layer pea coal
- 3 inch layer buckwheat coal
- 3 inch layer rice coal
- 3 inch layer barley coal
- 30 inch layer crushed anthracite of 20 mesh
- 30 inch freeboard from top of coal to limit of cylindrical portion of tank

the several layers being arranged to avoid channeling of liquid in the manner known to the art.

Such a filter will have a capacity of about 15 to 20 gallons per minute per square foot of bed area when the bath, which is introduced from the top, is subjected to a pressure of about 20 pounds per square inch. The bath is drained

from the bottom through suitable manifolds. Preferably a baffle is inserted near the top of the tank to avoid disturbance of the filter bed. In operation, the filter is used in a manner similar to a conventional gravel filter and may be cleaned by backwashing in the usual way. Especially good results are secured if the anthracite selected is that sold under the trade name "Anthrafilt," obtainable from the Anthracite Equipment Corporation, New York, New York.

If desired, the invention may be modified by deliberately increasing the concentration of the offensive anion, i. e., the ion tending to form an insoluble salt with the metal cations present, prior to the filtration step. This procedure is especially useful if the purification of the bath is expected in any way to increase the concentration of offensive cation.

For example, part of the bath may be subjected to an evaporation and crystallization step to reduce its water and sodium sulfate content and subsequently returned to the main body of the bath as mentioned above. The use of lead equipment in the evaporation step or the addition of sulfuric acid containing lead may increase the concentration of the offensive metal cation, or cation tending to form an insoluble salt with the offensive anion in the bath.

In such a case, the bath, prior to filtration, can be saturated with hydrogen sulfide under pressure, if desired, causing reduction in the concentration of the offensive cation and possibly precipitation of sulfur. It is then filtered carefully, also under pressure, if desired, and then aerated to remove the offensive sulfide anion. The bath will then be eminently suited for use.

For the removal of offensive sulfide anion, aeration has been proposed above. This procedure will remove hydrogen sulfide in part by mechanical displacement and in part by oxidation to sulfur. If the amount of sulfur liberated is excessive, the bath may be filtered again for clarification purposes. The offensive sulfide anion may be removed by mild oxidation reactions, such as by means of hydrogen peroxide, persulfuric acid, chlorine, electrolysis, etc., so long as the oxidizing agent does not introduce some other offensive cation into the bath.

The aeration or other oxidation of the bath is conveniently carried out in a suitably large open-top tank. When aeration is used the tank may be provided with perforated pipes near the bottom through which air may be blown. If air is introduced under a pressure of about 20 pounds per square inch, a flow of air in the ratio of about 1 cubic foot per minute for every 5-7 gallons per minute of bath flow will yield satisfactory results. If air under pressure of only 3-5 pounds per square inch is used, a ratio of about 1 cubic foot per minute of air for every 1.5-2.0 gallons per minute of bath flow will yield effective aeration. Such aeration is easily capable of lowering the reducing power of the bath (as hereinafter defined) from as much as 100 parts per million of sulfide ion to less than 10 parts per million.

The removal of offensive ions in the bath can be accomplished in other ways than those specifically described above. For example, cations, as well as anions, may be removed by electrolysis of the bath in a known manner, or by other means of precipitation and filtration. The offensive anion may be removed, as stated above, by an oxidation reaction other than aeration. In any method used, however, it is important that

other offensive ions are not thereby introduced into the bath.

Since the formation of these insoluble salts, such as lead sulfide, is dependent on the concentrations of the cations and anions in solution in the regenerating bath according to the chemical principle of mass action, their formation can be minimized, if not prevented, by maintaining the concentration of either the offensive, insoluble-salt-forming cations or anions or both so low that their solubility product is not exceeded in the vicinity of the spinneret face.

Similarly, it will be understood that if the concentration of an offensive cation, for example, is sufficiently low, the tolerable concentration of the offensive anion may be considerably higher than would be the case if the offensive cation concentration were high. The reverse situation is also true. Consequently, if both concentrations are maintained at as low a figure as possible, the chances of not exceeding the solubility product in the reaction zone at the face of the spinneret will be enhanced.

While it is preferable to keep both the lead and sulfide concentrations as low as possible, beneficial results are obtained when one is high, provided the other is low. Thus, if the total lead concentration is only 2 parts per million, a reducing power, calculated as a function of sulfide ion concentration, as high as 30 parts per million may be tolerated while, if the lead content is 6 parts per million, the reducing power should not exceed 10 parts per million. A total lead content as high as 8 parts per million may be tolerated if the sulfide concentration does not exceed about 7 parts per million measured as herein described. It has been found, as a general rule, that the product of the lead concentration in parts per million and the sulfide concentration in parts per million should be maintained less than 60.

The regenerating bath is, of course, preferably fed to the spinning machine at such a rate that the accumulated offensive sulfide ion concentration is less than that of a bath saturated with hydrogen sulfide until the bath has passed the last spinneret, or spinning position, and is ready for purification and recirculation. However, if no appreciable lead ion is picked up by the bath, little harm will obtain even if the bath becomes saturated with the offensive anion before the last spinning position is reached, for there will be insufficient offensive cation present to form appreciable amounts of insoluble salt in the reaction zone at the spinneret face and the benefits of the invention will still be enjoyed to a major extent. If the offensive metal cation content, particularly the lead content, of the bath is maintained at less than 4 parts per million without the introduction of other offensive metal ions very material diminution of deposition of insoluble foreign matter on the spinneret face will be accomplished. If each spinneret is provided continuously with fresh bath, as by means of the so-called "cross-flow" bath, i. e., a bath in which the bath is passed into the regenerating bath tank on the side of said tank where the spinnerets are positioned and removed on the opposite side of the tank, the pick-up of offensive ions can be minimized.

It will be readily apparent that it is not essential to remove the offensive ions from all of the coagulating bath. It will be sufficient if that part of the bath which is in contact with the spinneret is treated for removal of such offensive ions. The portion of the bath adjacent the spinnerets may, for example, be partitioned off from the re-

mainder of the bath and the offensive ions removed only from that portion of the bath which is in contact with the spinnerets. Alternatively, the two-bath system for coagulation of the filaments may be employed in which the offensive ions are removed only from the first bath.

The invention has been described in terms of the viscose process for the manufacture of rayon filaments involving the use of a regenerating bath in which the principal offensive cations and anions are lead and sulfide ions respectively. However, the invention is also applicable when other offensive ions are involved, such as cadmium, bismuth, copper, thallium, or even silver or mercury. Furthermore, if zinc ions, or other metal ions, are undesirable but necessarily present in the process, the concentration of the zinc, or other metal, can be controlled to obtain a reduction in cratering, caused by deposition of insoluble salts of these metals, in accordance with the present invention. Similarly, a different wet spinning process involving other offensive anions, such as sulfite, sulfate, carbonate, or the like will find the principles of this invention applicable.

Since it is obvious that many changes and modifications can be made in the above-described details without departing from the nature and spirit of the invention, it is to be understood that the invention is not to be limited to the details described herein except as set forth in the appended claims.

I claim:

1. The process of spinning regenerated cellulose filaments which comprises extruding viscose through a spinneret into an acid bath comprising a bath liquid containing lead ions and hydrogen sulfide, removing said bath liquid from said bath, saturating the bath liquid with hydrogen sulfide under pressure, filtering the bath liquid under pressure through an anthracite filter for the removal of lead sulfide from said bath liquid, and then removing hydrogen sulfide by aeration.

2. The process of spinning regenerated cellulose filaments which comprises extruding viscose through a spinneret into an acid bath comprising a bath liquid which contains heavy metal cations and anions which are capable of combining to form therein an insoluble metal salt and which bath liquid is subject to accumulation of said ions in an amount to exceed the solubility product of said ions to form said insoluble metal salt in the reaction zone at the face of the spinneret, removing said bath liquid from said bath, increasing the said anion content of the bath liquid to form said insoluble metal salt, filtering said insoluble metal salt from said bath liquid, then removing said anions from said bath liquid, and then returning said bath liquid back into said bath.

3. The process of spinning regenerated cellulose filaments which comprises extruding viscose through a spinneret into an acid bath comprising a bath liquid which contains lead ions and anions which are capable of combining to form therein an insoluble lead salt and which bath liquid is subject to the accumulation of said ions in an amount to exceed the solubility product of said ions to form said insoluble lead salt in the reaction zone at the face of the spinneret, removing said bath liquid from said bath, increasing the said anion content of the bath liquid to form said insoluble lead salt, filtering said insoluble lead salt from said bath liquid, then removing said anions from said bath liquid, and

then returning said bath liquid back into said bath.

4. The process of spinning regenerated cellulose filaments which comprises extruding viscose through a spinneret into an acid bath comprising a bath liquid which contains heavy metal cations and sulfide ions which are capable of combining to form therein an insoluble metal sulfide and which bath liquid is subject to the accumulation of said ions in an amount to exceed the solubility product of said ions to form said insoluble metal sulfide in the reaction zone at the face of the spinneret, removing said bath liquid from said bath, increasing the sulfide ion content of the bath liquid to form said insoluble metal sulfide, filtering said insoluble metal sulfide from said bath liquid, then removing sulfide ions from said bath liquid, and then returning said bath liquid back into said bath.

5. The process of spinning regenerated cellulose filaments which comprises extruding viscose through a spinneret into an acid bath comprising a bath liquid which contains lead ions and sulfide ions and which bath is subject to the accumulation of said ions in amounts exceeding the solubility product of said ions to form insoluble lead sulfide in the reaction zone at the face of the spinneret, removing said bath liquid from said bath, increasing the sulfide ion content of the bath liquid to form said insoluble lead sulfide, filtering said insoluble lead sulfide from said bath liquid, then removing sulfide ions from said bath liquid, and then returning said bath liquid back into said bath.

6. The process of spinning regenerated cellulose filaments which comprises extruding viscose through a spinneret into an acid bath comprising a bath liquid which contains lead ions and sulfide ions and which bath liquid is subject to the accumulation of said ions in amounts exceeding the solubility product of said ions to form insoluble lead sulfide in the reaction zone at the face of the spinneret, removing said bath liquid from said bath, passing hydrogen sulfide into said bath liquid to increase the sulfide ion content of said bath liquid and form insoluble lead sulfide, filtering said insoluble lead sulfide from said bath liquid, then removing hydrogen sulfide from said bath liquid, and then returning said bath liquid back into said bath.

7. The method of diminishing the deposition of insoluble metal salt on a spinneret in the spinning of regenerated cellulose filaments from viscose which is extruded into an acid spinning bath comprising a bath liquid containing anions and heavy metal cations capable of combining to form therein an insoluble metal salt and which bath is subject to the accumulation of said ions in an amount to exceed the solubility product of said ions to form said insoluble metal salt in the reaction zone at the face of the spinneret, the steps comprising removing the bath liquid from said bath, filtering said bath liquid in the presence of said accumulated anions to remove accumulated insoluble metal salt therefrom, then removing said accumulated anions from said bath liquid, and returning said bath liquid to said bath.

8. The method of diminishing the deposition of insoluble metal salt on a spinneret in the spinning of regenerated cellulose filaments from viscose which is extruded into an acid spinning bath comprising a bath liquid containing anions and lead ions capable of combining to form therein an insoluble lead salt and which bath is sub-

ject to the accumulation of said ions in an amount to exceed the solubility product of said ions to form said insoluble lead salt in the reaction zone at the face of the spinneret, the steps comprising removing the bath liquid from said bath, filtering said bath liquid in the presence of accumulated anions to remove said accumulated insoluble lead salt therefrom, then removing said accumulated anions from said bath liquid and returning said bath liquid to said bath.

9. The method of diminishing the deposition of insoluble metal salt on a spinneret in the spinning of regenerated cellulose filaments from viscose which is extruded into an acid spinning bath comprising a bath liquid containing sulfide ions and heavy metal cations capable of combining to form therein an insoluble metal sulfide and which bath is subject to the accumulation of said ions in an amount to exceed the solubility product of said ions to form said insoluble metal sulfide in the reaction zone at the face of the spinneret, the steps comprising removing the bath liquid from said bath, filtering said bath liquid in the presence of accumulated sulfide ions to remove accumulated insoluble metal sulfide therefrom, then removing accumulated sulfide ions from said bath liquid, and returning said bath liquid to said bath.

10. The method of diminishing the deposition of insoluble metal salt on a spinneret in the spinning of regenerated cellulose filaments from viscose which is extruded into an acid spinning bath comprising a bath liquid containing sulfide ions and lead ions capable of combining to form therein insoluble lead sulfide and which bath is subject to the accumulation of said ions in an amount to exceed the solubility product of said ions to form said insoluble lead sulfide in the reaction zone at the face of the spinneret, the steps comprising removing the bath liquid from

said bath, filtering said bath liquid in the presence of accumulated sulfide ions to remove accumulated lead sulfide therefrom, then removing accumulated sulfide ions from said bath liquid and returning said bath liquid to said bath.

11. The method of diminishing the deposition of insoluble metal salt on a spinneret in the spinning of regenerated cellulose filaments from viscose which is extruded into an acid spinning bath comprising a bath liquid containing hydrogen sulfide and heavy metal cations capable of combining to form therein insoluble lead sulfide and which bath is subject to the accumulation of said hydrogen sulfide and lead ions in an amount to exceed the solubility product of the sulfide ions and lead ions to form said insoluble lead sulfide in the reaction zone at the face of the spinneret, the steps comprising removing the bath liquid from said bath, filtering said bath liquid in the presence of accumulated hydrogen sulfide to remove accumulated insoluble lead sulfide therefrom, then removing accumulated hydrogen sulfide from said bath liquid, and returning said bath liquid to said bath.

12. The method as defined in claim 7 in which the said anions are removed by aeration of the bath liquid.

13. The method as defined in claim 8 in which the said anions are removed by aeration of the bath liquid.

14. The method as defined in claim 9 in which the sulfide ions are removed by aeration of the bath liquid.

15. The method as defined in claim 10 in which the sulfide ions are removed by aeration of the bath liquid.

16. The method as defined in claim 11 in which the hydrogen sulfide is removed by aeration of the bath liquid.

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