

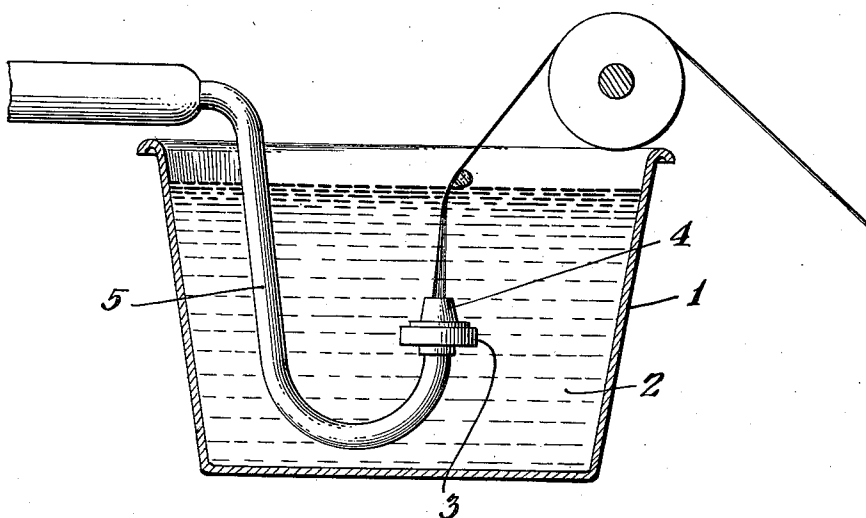
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SPINNING FIBERS

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SPINNING FIBERS

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The present invention relates to the manufacture of fibers, and more particularly to an improved process and apparatus to be used in the step of spinning the fibers to insure their production with improved and uniform characteristics.

An object of my invention is to improve the step of spinning artificial fibers so that the fibers produced are uniformly free from brittleness, have increased tensile strength and flexibility, and possess a softness which is comparable to that of the simulated natural fiber.

Another object of my invention is to improve the spinning of artificial fibers in such a manner that the fibers produced will be free from deleterious impurities which tend in time, and under ordinary conditions, to bring about an undesirable discoloration of the fibers.

A further object of my invention is to provide an apparatus for accomplishing the above objects, and which can be formed from inexpensive, easily workable material, whereby the same may be constructed in a convenient and economical fashion.

Still a further object of my invention is to provide a spinning apparatus in which the spinnerette comprises a metal from which it may be formed with great facility and having sufficient toughness whereby the spinnerette will not suffer abrasion at the orifices during the spinning operation, but which metal would dissolve in the fiber coagulating bath and injure the fibers were it not for the prevention of the dissolution of the metal and consequent harm to the fiber.

An additional object of my invention is to prevent dissolution in the fiber coagulating bath of a spinnerette made of a metal normally harmful to the fibers, by contacting the spinnerette in the fiber coagulating bath with a metal which dissolves in preference to the metal of the spinnerette, and which dissolving metal upon solution forms a salt which is less injurious to the fibers and which, preferably, is a desirable, or at least a harmless, ingredient of the coagulating bath.

My invention is illustrated in the accompanying drawing in which the single figure is a diagrammatic illustration partly in section and partly in elevation.

Although I will describe my invention more particularly with reference to the spinning of synthetic protein fibers made from casein, it will be apparent to one skilled in the art that my invention is applicable to, and includes within

its scope, the spinning of artificial fibers generally.

In accordance with the preferred method of my invention, the composition from which the fiber is made is prepared by dissolving casein, either from a vegetable source such as soya beans or an animal source such as milk, in a liquid with the assistance of a dispersing agent. The casein solution thus prepared and having a desired viscosity is extruded through fine orifices in a spinnerette immersed in an acidic coagulating bath. A substantial amount of sulfuric acid is contained in the bath, together with other reagents which advantageously may be present in the bath, such as sodium sulfate and the general class of casein hardening alums, i. e., potassium aluminum sulfate, and aluminum sulfate. The extruded casein solution, upon contact with the coagulating bath, receives an initial coagulation, thus producing filaments which may be continuously withdrawn from the bath. In order that the fibers may be completely and uniformly coagulated throughout their entire cross-section, it is necessary to permit the fibers to remain in the coagulating bath or to be kept wet with coagulating liquid that normally adheres to them for a substantial period of time.

The solution or dispersion of the casein that is extruded from the spinnerette into the coagulating bath is probably in the form of a caseinate, which upon contact with the acid is reconverted into casein. This involves an interaction between the spun fiber and the acids and hardening ingredients of the bath. The complete coagulation of the fiber requires the ingredients of the coagulating bath to react with or become intimately admixed with the fiber.

I have discovered that the casein solution to be extruded should be free from such heavy metals as iron, manganese, and particularly copper and their salts, because the presence of even minute amounts of these materials in the extruded fibers imparts undesirable characteristics to them. The copper present in ionized form should not exceed one part in one million and preferably one part in twenty million, while active iron should preferably be present in not over one part in two hundred thousand.

One of the deleterious effects of the presence of these impurities in the fiber, for example, is the rendering of the fiber undesirably brittle.

Fibers containing such heavy metals, when contacted with a sulfide, also exhibit an undesirable dark color characteristic of heavy metal sulfides, thus indicating that heavy metals have been de-

posited in the fibers. It will be appreciated that sulfides are not only present in the atmosphere as gases, but are found to a much greater extent in water and washing compounds. It is to be expected that fibrous material containing such heavy metals and/or their salts after a lapse of time and particularly upon laundering, will exhibit an undesirable discoloration.

I have also discovered that the presence of a small amount of the heavy metals or their salts in the coagulating bath has a similarly damaging effect upon the fibers being produced. It is believed that the prolonged and intimate contact between the filaments and the coagulating liquid for establishing uniform coagulation throughout the filaments necessarily causes impurities in the coagulating liquid to be deposited or otherwise retained within the fibrous mass, and that this occurs substantially as effectively as though the impurities were present in the original casein solution.

By a careful selection of pure ingredients and mixing the same under controlled conditions, it is possible to formulate an initial casein spinning solution which contains a very small or an insignificant amount of the undesirable impurities of the type discussed above. It is not so simple to employ a non-contaminating coagulating bath, because due to its acidic nature, the bath dissolves any contacting parts fabricated of heavy metal.

It has been usual heretofore, to form the tank containing the coagulating bath of some inert non-metallic material, such as stone or wood and the like. Attempts have also been made to fabricate spinnerettes from relatively inert metals such as tantalum, platinum or gold in order to prevent their solution, but such materials are generally avoided because such inert metals, as a rule, are extremely soft and often difficult to solder, with the result that a spinnerette of a desired design can be fabricated only with great difficulty. Another inherent disadvantage in the use of soft metals is that the abrasion of the spinnerette orifices by the extruded solution rapidly enlarges and distorts the orifices with the result that a uniform fiber and one of desired cross-sectional dimensions and form cannot be produced and the spinnerette must be frequently replaced. Moreover, such materials are relatively costly and greatly increase the expense involved in the process.

Materials which are inexpensive, relatively tough, and from which a spinnerette can be readily formed, such as brass, would be ideal from the manufacturing or economic point of view, as well as because a spinnerette formed of such metal would retain its orifice dimensions without appreciable abrasion by the extruded solution. I have observed, however, that when such apparatus is utilized in the step of spinning artificial fibers, the fibers being produced exhibit the characteristic brittleness of fibers containing undesirable metal impurities, and the presence of such impurities may be further evidenced by the sulfide test discussed above. Careful weighing of the spinnerette and supporting apparatus shows that they have suffered a decrease in mass which may be correlated to the undesirable results being obtained.

I have pointed out heretofore that in the case of spinning synthetic fibers from casein, there are certain metals, such as aluminum, which in the form of their salts are desirable addition agents in the coagulating bath. I have also dis-

covered that there are certain metals, such as zinc, which when present in the bath in the form of their salts, exert in some cases, an apparent beneficial effect upon the synthetic fibers produced, and in general have at least no appreciable detrimental effect. But aluminum and other soft metals such as, zinc, are not suitable for use in forming a spinnerette because the same would corrode rapidly, particularly at the orifices, with the result that frequent replacements would be necessitated.

It is apparent that the various metals may be evaluated and listed in accordance with the effects they produce upon the characteristics of the extruded fibers when present as salts in the coagulating bath. In the spinning of casein fibers, for example, the metals will range from those producing a decided harmful effect such as, copper, manganese, and iron, to those exerting a highly beneficial effect such as aluminum. The comparative rating to be given a particular metal will depend on the fibers being produced.

In accordance with my invention, I employ a spinnerette made from a metal which normally is readily workable and which is harmful in the bath, but I avoid contamination of the coagulating bath by electrically coupling the spinnerette with a member formed of a metal or metals which have a less harmful effect and, preferably, are a beneficial or at least a harmless ingredient in the bath, and which coupling metal dissolves in the bath rather than the metal of the spinnerette. In order to prevent the dissolving of metal of the spinnerette and to accomplish this at the expense of the solution of the metal forming the coupling member, it is necessary to have said member electropositive with respect to the spinnerette. Some confusion exists as to the proper terminology to be employed, and I prefer to designate as "electropositive" the member of the system which is anodic and tends to pass into solution.

The simplest form of my invention is in the use of an electric couple in which the metal forming the protecting member is selected from the group of metals which, because of their electrolytic potential values, are normally electropositive with respect to the spinnerette metal. The electrolytic potential relationship between the various metals may be readily ascertained by reference to any standard table. Aluminum, magnesium and zinc are particularly desirable for forming the protecting member inasmuch as they are all relatively highly positive with respect to hydrogen. The selection of a particular metal will depend, of course, on the fiber being produced.

It will be apparent that when the spinnerette metal is selected from a lower position in the electrolytic potential table, the group of metals which may be used for protective purposes correspondingly increases.

In a particular embodiment of my invention as applied to the spinning of synthetic protein fibers from casein, a brass spinnerette may be coupled to an aluminum supporting bracket with satisfactory results. Brass is selected as the spinnerette material because of the ease with which the same can be fabricated, as well as its resistance to abrasion at the orifices thereof. Aluminum is selected as the protecting material because its presence in the coagulating fluid is considered to be beneficial, and, secondly, it has an electrolytic potential of 1.35 volts, as compared to the potential of the principal compo-

ment of brass, namely, -0.34 volt for copper, (all potentials referred to hydrogen as zero). As a result of these potential differences, the spinnerette metal does not appreciably pass into solution. Under certain conditions, a zinc supporting bracket may be substituted for the aluminum bracket described above with satisfactory results. The zinc couple effectively prevents the solution of copper from the spinnerette.

In the above embodiment, the couple has been described as one in which the aluminum or zinc is present in the form of a supporting bracket for the spinnerette, but it is to be understood that a couple in this form is merely for convenience, and that, if desired, the protecting member may be constructed independently of all or part of the apparatus used in spinning fibers. It is essential only that the protecting member be in contact with the coagulating liquid and in contact with or otherwise electrically connected to the spinnerette. An advantage in forming the protecting member independently of the spinning apparatus, is that the utilization of an implied E. M. F. as disclosed hereinafter is facilitated.

If desired, the spinnerette may be plated with a metal which is less reactive with an acidic coagulating bath than is the metal from which the spinnerette is formed. In the case of a brass spinnerette, for example, the same may be conveniently plated with nickel, gold or silver by the electrodeposition process. In the case of gold or silver, the same may be amalgamated to include mercury within the protecting coating. A metal such as nickel or mercury is not reactive with an acid coagulating bath probably because of its normal passivity or because of the normal high overvoltage of either oxygen or hydrogen when plated out thereon. This high overvoltage effectively counteracts the electrolytic potential normal to these metals. The protective value of a plate formed of noble metal, such as gold, is the result of the well known chemical inertness of these metals. The protecting member couple, discussed above, is utilized in combination with a plated spinnerette, primarily for preventing contamination of the acid coagulating bath by the exposed portions of the metal from which the spinnerette is formed, particularly at the orifices thereof.

In the drawing the coagulating bath 2 is contained in a tank 1, in which coagulating bath a spinnerette 4 is immersed. The metal of the spinnerette 4 is cathodic with respect to the protecting member 3 or the support 5, or both, as has been described heretofore.

A further variation in the embodiment of my invention is to separate the spinnerette from the protecting member so that a small potential from an outside source may be impressed across the spinnerette and the member. This potential may be utilized to augment a normal positive potential of the protecting member with respect to the spinnerette due to their electrolytic potential values, or to cause the protecting member to be anodic with respect to said spinnerette where the electrolytic potential values are substantially equal or the spinnerette is normally anodic relative to the protecting member.

It will be appreciated that in forming the coagulating solution or the casein solution from materials of ordinary commercial purity, the solutions will almost always contain initially a small but undesirable percentage of copper and iron or other heavy metals. It is an advantage

of my invention that the protecting member will also act to purge the solutions of these minute quantities of undesirable heavy metals by electrolytic deposition i. e., the impurities will plate out and be replaced by the metal forming the protecting member. The purging effect with respect to the casein solution is more pronounced when the protecting member contacts the solution prior to its extrusion and coagulation into synthetic fibers.

It is understood that the use of electric couples and the like is well known in the electrochemical art, but there has been no appreciation of the fact that electrochemical principles could be applied to the art of spinning artificial fibers to prevent the formation of ions in the coagulating bath which adversely affect the characteristics of the fibers being produced, in favor of the formation of ions which have a harmless or beneficial action.

Variations in my invention such as the metals to be employed, and their arrangement, will depend upon the fiber being formed and details of the spinning process, and are to be considered within the scope of the invention as defined in the following claims.

I claim:

1. An apparatus for spinning synthetic fibers comprising a spinnerette formed of a metal which normally dissolves in an acidic coagulating liquid with deleterious effects upon synthetic fibers coagulated therein, and a protecting member electrically connected to the spinnerette and formed of a metal which upon solution in an acidic coagulating liquid has a less deleterious effect upon synthetic fibers coagulated therein than that resulting from the solution of the spinnerette metal, said protecting member being anodic with respect to said spinnerette, whereby during contact of the spinnerette and the protective member with an acidic coagulating bath, the protective member dissolves instead of the spinnerette.

2. An apparatus for spinning synthetic fibers comprising a spinnerette formed of a metal which normally dissolves in an acidic coagulating liquid with deleterious effects upon fibers being coagulated therein, and a protecting member electrically connected to said spinnerette and formed of a metal which exerts a less deleterious effect when dissolved in an acidic coagulating liquid upon the fibers being coagulated therein than that resulting from the solution of the spinnerette metal; the metal of the protecting member being anodic with respect to the spinnerette metal whereby it dissolves instead of the spinnerette when the spinnerette and the protective member are in contact with an acidic coagulating bath.

3. An apparatus for spinning synthetic protein fibers which are harmed during the spinning operation by contact with a solution containing certain heavy metals, comprising a container for holding an acidic solution containing a metal salt that exerts a beneficial action in the coagulating of the fibers, a spinnerette within said container adapted to be immersed in the acidic coagulating solution and formed of one of said heavy metals which normally dissolve in the coagulating solution to exert said harmful action upon the protein fibers being produced, and which resist abrasion during the spinning operation, and a protecting member also within the container electrically connected to said spinnerette and also adapted to contact the coagulating liquid, said protecting member being formed of

a metal which upon dissolving in the coagulating solution forms a salt that is present in said solution and exerts said beneficial action upon the fibers, said metal of the protecting member also being anodic with respect to the metal of said spinnerette whereby it dissolves in preference to the metal spinnerette.

4. An apparatus for spinning synthetic protein fibers in an acidic coagulating bath, comprising a brass spinnerette and an aluminum protecting member electrically connected thereto, said spinnerette and protecting member being adapted to be immersed in said acidic coagulating bath during the spinning operation, whereby the aluminum dissolves in the bath in preference to the brass.

5. Apparatus for spinning synthetic fibers in an acid coagulating bath containing an aluminum salt, comprising a brass spinnerette and an aluminum supporting bracket therefor electrically connected to the spinnerette, said spinnerette and support being adapted to be immersed in said acidic coagulating bath during the spinning operation, whereby the aluminum dissolves in the bath in preference to the brass.

6. An apparatus for spinning synthetic fibers in an acidic coagulating bath, comprising a spinnerette formed of copper-containing metal, and a metallic protecting member electrically connected thereto, said protecting member being anodic with respect to copper whereby the metal of said protecting member dissolves in the bath in preference to the copper.

7. In a method of spinning artificial fibers from material that is extruded through orifices of a spinnerette into an acidic coagulating bath, which material is harmed by contact with a coagulating bath containing certain heavy metals in solution, the prevention of contamination of the bath by a spinnerette formed of at least one of said harmful metals which is in contact with the acidic bath by maintaining a protecting member electrically connected to said spinnerette and in contact with the bath, which pro-

tecting member is formed of metal which upon solution in the acidic coagulating bath has a less deleterious effect upon the fibers than that produced by a solution of the harmful metal in the spinnerette and which protecting member is anodic with respect to said spinnerette, whereby the coagulating bath tends to dissolve the metal of the protecting member in preference to the metal of the spinnerette.

8. In a method of spinning artificial fibers from material that is extruded through the orifices of a spinnerette into an acidic coagulating bath, which material is harmed by contact with a coagulating bath containing certain heavy metals in solution, the prevention of contamination of said bath by a spinnerette formed of at least one of said harmful metals which is in contact with the acidic bath by maintaining a protecting member electrically connected to said spinnerette and in contact with the bath, which protecting member is formed of metal which upon solution in the coagulating bath has a less deleterious effect upon the fibers than that produced by a solution of the harmful spinnerette metal, and which also is anodic with respect to the metals of said spinnerette, whereby the coagulating bath tends to dissolve the metal of the protecting member in preference to the metal of the spinnerette.

9. In a method of spinning synthetic protein fibers from a casein solution that is extruded through a brass spinnerette into an acidic coagulating bath containing aluminum salts, which solution is harmed by contact with a coagulating bath containing dissolved copper, the prevention of contamination of said bath by the brass spinnerette immersed in said acid by maintaining the brass spinnerette in electrical contact with an immersed aluminum protecting member, whereby the coagulating bath tends to dissolve the aluminum of the protecting member in preference to the brass of the spinnerette.

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