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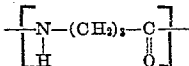
COMPOSITION COMPRISING POLYPYRROLIDONE POLYMER DISSOLVED IN FERRIC CHLORIDE SOLUTION AND PROCESS FOR PREPARING SAME

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This invention relates to new compositions of matter. More particularly, the invention relates to new compositions of matter comprising polypyrrolidone and solvents therefor.

As used in this invention the term "polypyrrolidone" is intended to include a useful high molecular weight thermoplastic product which is produced by the polymerization of 2-pyrrolidone and has become known as polypyrrolidone, having in recurring relationship the following chemical structural unit:



Polypyrrolidone possesses many excellent properties which make it desirable for utilization in the manufacture of end products, such as ribbons, films, fibers, filaments, rods, bristles, lacquers, coatings, shaped articles and the like. Polypyrrolidone can be converted into shaped articles in many ways. For example, it may be cast into films or forced through multi-hole spinnerets to form fibers or filaments. Regardless of the end use to which the polypyrrolidone is to be put, it is generally more convenient and efficient to employ the polymer in a solution. This is well illustrated in the textile industry where polypyrrolidone is employed in the formation of fibers and filaments, which are manufactured by several methods of spinning, such as melt spinning, dry spinning and wet spinning.

In the melt spinning method, the polymer is heated to a high temperature until it becomes molten, and is thereafter forced through filter media, and thence through a spinneret from whence it is extruded in filamentary form. This method has, however, many disadvantages, although it is widely used in the industry at the present time in the production of synthetic fibers and filaments. The high temperatures used in melt spinning require the exercise of extreme care in order to prevent the decomposition of the polymer. Furthermore the high temperatures also affect the physical and chemical characteristics of the polymer and thereby result in a product of inferior quality. In addition to these disadvantages, it is extremely difficult to add to the molten polymer at such high temperatures compounds such as dyes, anti-static agents, light stabilizers, and the like.

In the dry spinning method of fiber formation, the polymer is dissolved in a suitable solvent and subsequently extruded from spinnerets into a heated atmosphere in order to evaporate the solvent. Even this method, however, has its disadvantages, since during the period of time in which the solvent evaporates, considerable damage may be inflicted on the fibers because of the high heat necessary to bring about solvent evaporation. Another disadvantage of the dry spinning method, and of the melt spinning method also, is the added cost necessary to maintain such high temperatures needed to manufacture the desired end product.

The wet spinning method obviates many of the disadvantages of both melt spinning and dry spinning. In order to form filaments by the wet spinning method, the polymer is dissolved in a suitable solvent and extruded from a spinneret into a coagulating bath capable of leach-

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ing the solvent from the fibers. Normally, this method may be carried out at temperatures much lower than either the melt spinning or the dry spinning methods. If it is desired to use additives, such as dyes, anti-static agents, fire-retarding agents, light stabilizers and the like, in the polymeric solution, they may be incorporated therein without the danger of decomposition or seriously affecting the properties of the end product where the wet spinning method of filamentary formation is employed. It is much easier to introduce such additives into a solution than to introduce them into a molten composition. Then again, solutions are much easier to handle during processing, and in many cases may be stored for long periods of time without a change of physical and chemical properties. It is much easier to cast a film from a solution than to cast it from a molten composition. It is readily apparent, therefore, that solutions of polypyrrolidone possess many distinct advantages over molten compositions in the manufacture of end products.

Accordingly, it is a primary object of the present invention to provide new and useful compositions of matter comprising polypyrrolidone. It is a further object of the invention to provide solutions of polypyrrolidone which may be converted into shaped articles, such as ribbons, films, filaments, rods, fibers, bristles, and the like. It is still another object of the invention to provide a process for the preparation of clear, viscous, spinnable solutions from polypyrrolidone polymer and a solvent which is non-corrosive and easily handled.

In general, the objects of the invention are accomplished by dissolving polypyrrolidone in a solution of ferric chloride to form a homogeneous mixture. In the practice of this invention, ferric chloride is normally employed only in solution and not in its normal solid crystalline form. Suitable compounds which may be utilized to form a solution of ferric chloride comprise water, saturated aliphatic alcohols containing from 1 to 2 carbon atoms, and halogenated saturated aliphatic alcohols containing 2 carbon atoms. As examples of such alcohols there may be named methanol, ethanol, ethylene glycol, 2-bromo-ethanol, 2-chloro-ethanol, 2,2-dichloro-ethanol, 2-fluoro-ethanol, and 2,2,2-trichloroethanol. It has been found, contrary to expectations, that higher alcohols, such as propanol, t-butanol and butenediol, when used to form a solvent solution of ferric chloride, render the solvent solution ineffective as a solvent for polypyrrolidone. The ferric chloride is usually employed in a range of 25 to 40 percent based on the total weight of the solvent solution. The invention is practiced at room temperature or at slightly elevated temperature, depending on the nature of the polymer and the concentrations of polymer and solvent solution employed.

It will be readily apparent to those skilled in the art that polypyrrolidone can be dissolved in the solvent solution of this invention in widely varying concentrations. When the solution is to be used in the manufacture of fibers and filaments it is preferred to employ from about 15 to 25 percent polymer, based on the total weight of the polymer solution. From about 10 to 30 percent of the polymer, based on the total weight of the polymer solution, may be dissolved in the solvent solution of this invention when the polymer solution is to be used for other purposes, such as a coating or a lacquer and the like, or when lower or higher molecular weight polypyrrolidones are dissolved in the solvent solution.

The solvent solution of this invention readily dissolves polypyrrolidone within a wide range of temperature, depending upon the nature of the polymer and the concentration thereof to be dissolved in the solvent solution. Although temperatures within a range of 25° C. to 75° C. are preferred as a practical matter in bringing about solution, temperatures as low as 20° C. and up to the boil-

ing point of the polymer solution may be employed to bring about dissolution.

Polypyrrolidone soluble in the solvent solution of this invention may be prepared by various processes. Generally, however, polymeric pyrrolidone is prepared by polymerizing 2-pyrrolidone in the presence of a catalyst or a catalyst and an activator at a temperature in a range of -70°C. to 100°C. , with a range of 20°C. to 70°C. preferred. Polymer prepared in the presence of both a catalyst and an activator has greatly improved properties over polypyrrolidone prepared in the presence of a catalyst alone, accordingly polymer prepared in the presence of both is preferred. Catalysts that may be used in the preparation of polypyrrolidone include the alkali metals, namely, sodium, potassium and lithium, as well as the hydrides, hydroxides, oxides and salts of the alkali metals. Organic metallic compounds, preferably those which are strongly basic such as sodium phenyl, may be used as catalysts also. Activators such as the acyl compounds, lactones and alkyl esters of carboxylic acids, are usually employed. Simple polymerization methods may be used in the preparation of polypyrrolidone. It can be prepared readily by well-known solution, emulsion, suspension or bulk polymerization procedures. The solution and emulsion polymerizations may be either batch, semi-continuous or continuous methods.

The polypyrrolidone compositions which are useful in the practice of the present invention have a wide range of molecular weight, from about 3500 to 500,000. The preferred molecular weight range for the preparation of fibers is from 8500 to 50,000. Polymers of lower molecular weight may be used for coatings and films and higher molecular weight polymers may be used for molding shapes and similar articles. Specific viscosity of polypyrrolidone with a molecular weight of from about 3500 to 500,000 is in a range of from about 0.2 to 9.4 or more. Viscosity determinations are made by allowing a solution containing a 0.5 percent concentration of the polypyrrolidone in 90 percent formic acid to flow by gravity at 25°C. through a capillary viscosity tube.

The following examples are intended to illustrate the new compositions of this invention more fully but are not intended as limitative, as many widely varying modifications thereof are possible. In the examples all parts and percents are by weight unless otherwise indicated.

Example I

1 gram of polypyrrolidone, having a specific viscosity of 0.9, was easily dissolved in 2 grams ferric chloride hexahydrate and 2 grams of water at room temperature (25°C.) to give a clear solution of moderate viscosity. The solution was suitable for the production of fibers and films by wet or dry spinning methods.

Example II

1 gram of polypyrrolidone, having a specific viscosity of about 0.6, was easily dissolved in 2 grams ferric chloride hexahydrate and 2 grams absolute ethanol at room temperature (25°C.) to give a clear, viscous solution suitable for the production of fibers and films.

Example III

1 gram of polypyrrolidone, having a specific viscosity of about 0.6, was easily dissolved in 2 grams ferric chloride hexahydrate and 2 grams ethylene chlorohydrin at 75°C. to give a clear, viscous solution suitable for the production of fibers and films.

Polypyrrolidone polymers having widely varied molecular weights give like results when dissolved in the solvent solution of this invention.

As many widely apparently different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that the same is not to be limited to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. A new composition of matter comprising polypyrrolidone dissolved in a solvent solution comprising ferric chloride dissolved in a compound taken from the group consisting of water, saturated aliphatic alcohols containing from 1 to 2 carbon atoms, and halogenated saturated aliphatic alcohols containing 2 carbon atoms.

2. The composition defined in claim 1 wherein the solvent solution comprises ferric chloride dissolved in water.

3. The composition defined in claim 1 wherein the solvent solution comprises ferric chloride dissolved in ethanol.

4. The composition defined in claim 1 wherein the solvent solution comprises ferric chloride dissolved in ethylene chlorohydrin.

5. The composition defined in claim 1 wherein the solvent solution comprises ferric chloride dissolved in ethylene glycol.

6. A new composition of matter comprising from 10 to 30 percent based on the total weight of the composition, of polypyrrolidone dissolved in 70 to 90 percent, based on the total weight of the composition, of a solvent solution comprising 25 to 40 percent, based on the total weight of the solvent solution, of ferric chloride and 60 to 75 percent, based on the total weight of the solvent solution, of a compound taken from the group consisting of water, saturated aliphatic alcohols containing from 1 to 2 carbon atoms, and halogenated saturated aliphatic alcohols containing 2 carbon atoms.

7. The composition defined in claim 6 wherein the solvent solution comprises ferric chloride dissolved in water.

8. The composition defined in claim 6 wherein the solvent solution comprises ferric chloride dissolved in ethanol.

9. The composition defined in claim 6 wherein the solvent solution comprises ferric chloride dissolved in ethylene chlorohydrin.

10. The composition defined in claim 6 wherein the solvent solution comprises ferric chloride dissolved in ethylene glycol.

11. A new fiber forming composition of matter comprising from 15 to 25 percent, based on the total weight of the composition, of polypyrrolidone dissolved in 75 to 85 percent, based on the total weight of the composition, of a solvent solution comprising 25 to 40 percent, based on the total weight of the solvent solution, of ferric chloride and 60 to 75 percent, based on the total weight of the solvent solution, of a compound taken from the group consisting of water, saturated aliphatic alcohols containing from 1 to 2 carbon atoms, and halogenated saturated aliphatic alcohols containing 2 carbon atoms.

12. The composition defined in claim 11 wherein the solvent solution comprises ferric chloride dissolved in water.

13. The composition defined in claim 11 wherein the solvent solution comprises ferric chloride dissolved in ethanol.

14. The composition defined in claim 11 wherein the solvent solution comprises ferric chloride dissolved in ethylene chlorohydrin.

15. The composition defined in claim 11 wherein the solvent solution comprises ferric chloride dissolved in ethylene glycol.

16. A process for preparing a new composition of matter comprising mixing polypyrrolidone and a solvent solution comprising ferric chloride dissolved in a compound taken from the group consisting of water, saturated aliphatic alcohols containing from 1 to 2 carbon atoms and halogenated saturated aliphatic alcohols containing 2 carbon atoms, and heating the mixture to a temperature in a range of 20°C. to the boiling point of the mixture to form a homogeneous solution.

17. The process defined in claim 16 wherein the sol-

vent solution comprises ferric chloride dissolved in water.

18. The process defined in claim 16 wherein the solvent solution comprises ferric chloride dissolved in ethanol.

19. The process defined in claim 16 wherein the solvent 5

solution comprises ferric chloride dissolved in ethylene chlorohydrin.

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