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METHOD AND APPARATUS FOR PRECIPITATING CELLULOSE ACETATE

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11 Claims. (Cl. 18—51)

This invention relates to method and apparatus for precipitating cellulose acetate.

The acetylation of cellulose by one well-known method results in a viscous solution of cellulose acetate in acetic acid. Before this cellulose acetate is further processed to produce films, filaments or other products it is usually separated from the acetic acid solvent by precipitation and washing. This precipitation is brought about by diluting the solvent with water; in one case by adding water slowly to the so-called acid dope; in another case by running the dope into a bath of water or very dilute acid. When precipitating by the latter method, it is impossible with ordinary means of agitation to obtain a uniformly sized precipitate of the cellulose acetate. The fines are a source of loss during washing, and the larger particles require grinding to adapt the same for further processing. Further fines resulting from the grinding cause a further loss.

On the other hand, the larger particles formed in the precipitation are only slowly freed from acid in the washing process and cause this washing operation to be retarded.

It is therefore the principal object of the present invention to control the size of the precipitate to produce uniform particles. Other objects may present themselves as the following description proceeds.

In the drawing—

Figure 1 is a vertical section through apparatus for precipitating cellulose acetate according to the present invention;

Figure 2 is an inverted plan of the nozzle and knife employed; and

Figure 3 is a vertical section through the same.

The precipitating bath is contained in a precipitating chamber 5, and the cellulose acetate acid dope is supplied thereto from a supply tank 4, preferably supported by a bracket 6 on the chamber 4. The supply tank 4 has a removable cover 7 to permit charging thereof, and an air pipe 8 to supply compressed air thereto after charging.

From the tank 4, a supply pipe 9 extends down into the chamber 5. The lower end of the pipe 9 receives a cap 10 containing an extrusion orifice 12, and forming an extrusion nozzle.

Cooperating with the nozzle is a knife 14 keyed to the lower end of a vertical shaft 15 journaled in bearing brackets 16 on the tank 4 and pipe 9. The upper end of the shaft 15 carries a pulley 17 connected by a belt 18 to a pulley 19 on a shaft 20 journaled in a bearing 21 in a bridge 22 supported by the chamber 5. The upper end of the shaft 20 is suitably journaled and driven by any desired source of power, while the lower end has keyed thereto an agitator blade 23.

In operation, water or dilute acid is supplied to the chamber 5, while cellulose acetate acid dope is charged into the tank 4. Compressed air is introduced through the pipe 8, and forces the dope through the pipe 9 and out through the orifice 12.

To this, of course, obvious that a pump could be used for transferring the acid dope from the chamber 4 to the pipe 8 and orifice 12 without changing the nature of this invention. The precipitating liquid in the chamber 5 contacts the stream or rod 24 of acid dope thus extruded from the orifice 12, and precipitates the same.

At the same time, the knife 14 is rotated at a predetermined rate to interrupt the stream at regular intervals by cutting the same into uniform lengths or pellets 25 of uniform size. Meanwhile, the rotation of the blade 23 agitates the precipitating bath, and carries away the pellets as formed, and also carries away the acid or other products of the precipitating reaction, preventing local concentration of these about the nozzle 25 and knife.

The precipitating bath is maintained at a level well above the orifice 12. It is obvious that the rate at which the shaft 15 is to be driven, to give the size of particle desired, may be readily calculated from the dimensions of the blade 14 and the extrusion flow rate, in turn dependent upon the extrusion air pressure, viscosity of the dope and area of the orifice 12. A particle size of one eighth of an inch in both length and diameter is preferred, but may be larger or smaller as desired.

The uniform sized particles resulting from this process are washed and further processed preferably for the production of cellulose acetate rayon, but may be employed for other products.

The invention embraces such embodiments of the disclosed ideas as fall within the scope of the appended claims.

1 claim:

1. Method of precipitating cellulose acetate from acid dope formed by acetylation of cellulose, which comprises extruding a stream of said acid dope below the surface of a bath of dilute aqueous precipitating liquid, and simultaneously shearing the precipitate into pellets of uniform size.

2. Method of precipitating cellulose acetate from acid dope to facilitate washing and drying preparatory to further processing to produce films, filaments or other products, which comprises ex—
truding a rod like stream of constant diameter many times greater than the thickness of said films or filaments, and shearing said stream at regular intervals to produce pellets of uniform size and shape.

3. Method of precipitating cellulose acetate from acid dope formed by acetylation of cellulose which comprises extruding a stream of said acid dope below the surface of a bath of dilute aqueous precipitating liquid and simultaneously shearing the precipitate into pellets and agitating said liquid, said extrusion and shearing rates being in timed relation to produce uniform size of pellets, and said agitation rate also being in timed relation thereto to carry away said pellets and the acid formed by said precipitation to prevent local concentration of the products of the precipitation.

4. Method of precipitating cellulose acetate from cellulose acetate acid dope to facilitate washing and drying of the precipitate preparatory to dissolving the same in acetone to form the spinning or film casting solution, which comprises extruding below the surface of a bath of dilute aqueous precipitating liquid a stream of said cellulose acetate acid dope of constant cross sectional area over a hundred times that of rayon filaments, whereby contact of said dope and liquid causes precipitation of said cellulose acetate, and mechanically controlling the length of the precipitate simultaneously with the precipitation step by shearing the extruded material at regular intervals as it is being precipitated, to cut the same into successive pellets of uniform size and shape.

5. Apparatus for precipitating cellulose acetate from cellulose acetate acid dope to facilitate washing and drying of the precipitate preparatory to dissolving the same in acetone to form the spinning or film casting solution, comprising in combination means for extruding below the surface of a bath of dilute aqueous precipitating liquid a stream of said cellulose acetate acid dope of constant cross sectional area over a hundred times that of rayon filament whereby contact of said dope and liquid causes precipitation of said cellulose acetate, and means for mechanically controlling the length of the precipitate simultaneously with the precipitation step by shearing the extruded material at regular intervals as it is being precipitated, to cut the same into successive pellets of uniform size and shape.

6. Apparatus for precipitating cellulose acetate from acid dope formed by acetylation of cellulose, comprising in combination a nozzle, an acid dope supply tank in communication with said nozzle, a chamber containing a bath of dilute aqueous precipitating liquid, means for supporting said nozzle below the surface of said bath, a knife adjacent said nozzle, a shaft for driving said knife to move its cutting edge across the orifice of said nozzle, a bearing for said knife shaft, means attached to said nozzle supporting means for rigidly supporting said bearing and means for forcing dope from said tank through said nozzle into said liquid and into engagement with said knife, a shaft for driving said knife, a bearing for said knife shaft, means attached to said nozzle supporting means for rigidly supporting said bearing and means for forcing dope from said tank through said nozzle into said liquid and into engagement with said knife, an agitator having a blade below the surface of said bath, a shaft for driving said agitator, a bearing for said agitator shaft attached to said chamber, and means for driving said shafts in timed relation from a single source of power.

8. Method of precipitating cellulose acetate from cellulose acetate acid dope in a form to facilitate washing and drying of the precipitate preparatory to dissolving the same in acetone to form a solution for extrusion and coagulation, which comprises extruding below the surface of a bath of dilute aqueous precipitating liquid a stream of said cellulose acetate acid dope of constant cross sectional area over a hundred times that of a rayon filament whereby contract of said dope and liquid causes precipitation of said cellulose acetate, and mechanically controlling the length of said precipitate simultaneously with the precipitation step by shearing the extruded material at regular intervals approximately equal to the greatest dimension of its cross sectional area as it is being precipitated, to cut the same into pellets of uniform size and shape.

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