Dec. 23, 1952 G. T. DETWILER 2,622,273

METHOD AND MEANS FOR PRECIPITATING CELLULOSE ACETATE

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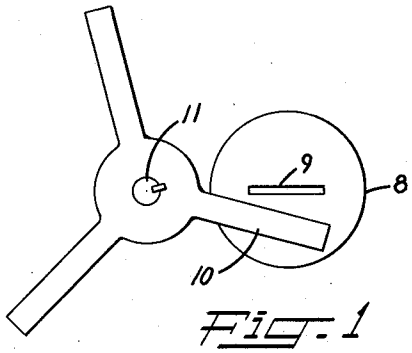
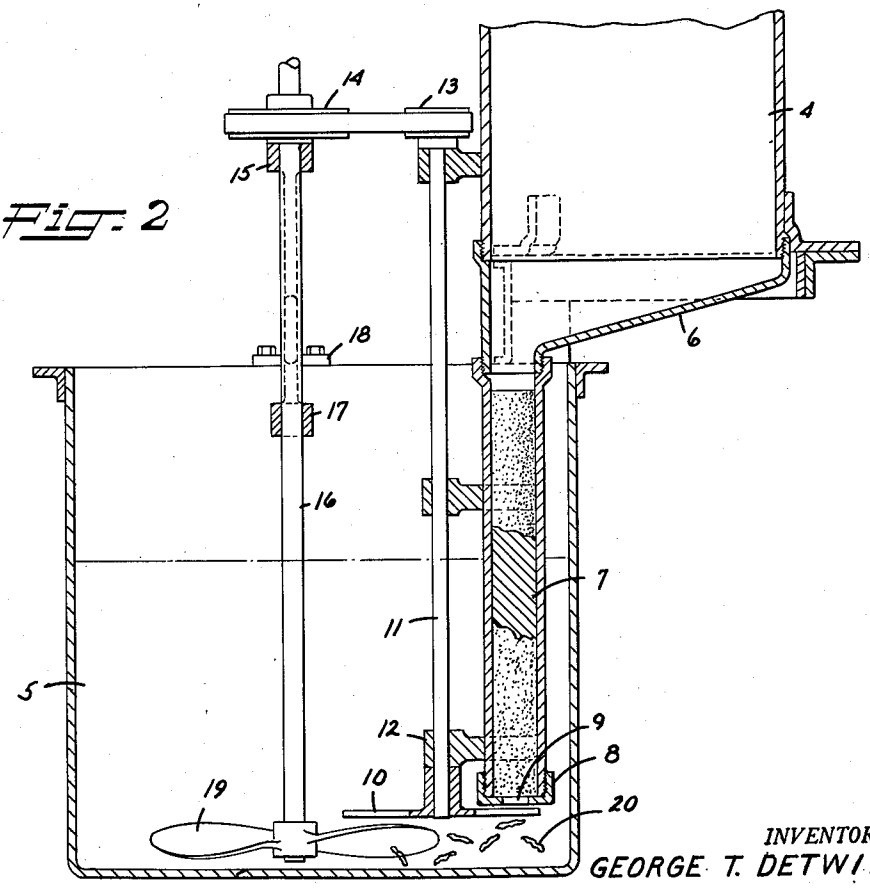


Fig. 1

Fig. 2



INVENTOR.
GEORGE T. DETWILER
BY
Thomas R. O'Malley

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METHOD AND MEANS FOR PRECIPITATING CELLULOSE ACETATE

George T. Detwiler, Chester, Pa., assignor to American Viscose Corporation, Wilmington, Del., a corporation of Delaware

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2 Claims. (Cl. 18—3)

1

This invention relates to a method and apparatus for precipitating cellulose acetate in the form of thin, substantially flat, porous particles.

The acetylation of cellulose by one method results in a viscous solution of cellulose acetate in acetic acid. Before this cellulose acetate is further processed to produce films, filaments, and 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.

It has been proposed to extrude the acid dope into the precipitating bath through an orifice of circular cross-section and shear the rod-like stream of extruding dope into segments of uniform size. That procedure results in the production of a precipitate in the form of comparatively thick, smooth, dense pellets which are not readily penetrated by fluids and require a considerable time for washing, drying and dissolving in the spinning or casting solvent.

It is a primary object of the present invention to produce a cellulose acetate precipitate in a faster washing, faster drying and faster dissolving form. This and other objects of the invention are accomplished by extruding the cellulose acetate into the precipitating bath through an elongated slot, in the form of a substantially flat stream and contacting the flat stream with a cutting means so positioned that the cutting edge thereof strikes against the face of the extruding stream to separate it into flat sections.

The invention will be readily understood when read in the light of the following description having reference to the drawing, in which,

Fig. 1 is an inverted plan of the elongated extrusion slot and knife employed to separate the stream; and

Fig. 2 is a vertical section through apparatus for precipitating cellulose acetate using an elongated extrusion slot in accordance with the present invention.

The precipitating bath is contained in a precipitating chamber 5 and the cellulose acetate dope is supplied thereto from a supply tank 4, preferably supported by a bracket 6 on the chamber 5. The supply tank 4 has a removable cover (not shown) to permit charging thereof and an air pipe (not shown) to supply compressed air thereto after charging. From the tank 4 the supply pipe 7 extends down into chamber 5. The lower end of pipe 7 receives a cap 8 containing an extrusion elongated slot 9 and forming an extrusion nozzle. Cooperating with the nozzle is a knife 10 keyed to the lower end of a vertical shaft 11 journaled in bearing brackets 12 on the tank 4 and pipe 7. The upper end of the shaft 11 carries a pulley 13 connected by a pulley 14 to a pulley 15 on the shaft 16 journaled in a bearing 17 in a bridge 18 supported by the chamber 5. The upper end of the shaft 16 is suitably journaled and driven by any desired source of power while the lower end is keyed to an agitator 19.

2

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 into chamber 4, and forces the dope through the pipe 7 and out through the extrusion slot 9.

It is obvious that a pump could be used for transferring the acid dope from the chamber 4 to the pipe 7 and slot 9 without changing the nature of this invention.

The precipitating liquid in the chamber 5 contacts the flat stream of acid dope leaving the slot 9 and precipitates the cellulose acetate.

At the same time, i. e. simultaneously with extrusion of the dope, the knife 10 is rotated at a predetermined rate so that its cutting edge intermittently strikes against the face of the flat stream of dope leaving the slot 9 at regular intervals and cuts it into flat sections. The knife striking against the face of the flat stream tends to attenuate and draw out the individual sections to produce thin, substantially flat particles 20 of irregular contour. The rotation of the blade 19 agitates the precipitating bath and carries away the particles as they are formed, and also carries away the acid or other products of the precipitating reaction preventing local concentration of these around the nozzle and knife. The precipitating bath is maintained at a level well above the slot 9.

Obviously the rate at which the shaft 11 is driven to give the size of particle desired may be readily calculated from the dimensions of the blade 10 and the extrusion flow rate, in turn dependent upon the air pressure, viscosity of the dope, and the area of the slot. The elongated slot may have a length of from ½ to 1½" and a width of from ⅛ to ¼". A slot having the dimensions .065" x .750" (area equals .0489 sq. in.) is entirely satisfactory.

By the action of the precipitating liquid, the attenuated sections of dope are set in the form of thin, substantially flat cellulose acetate particles which are characterized by a rough, crepe-

like surface, and porosity. These rough, porous particles can be washed, dried and dissolved more quickly than can the smooth, dense pellets which are obtained when the dope is extruded through an orifice of circular cross-section. For example when cellulose acetate dope was extruded through an elongated slot having an area of .0489 sq. in. into a 12 to 12.5% acetic acid precipitating bath over a period of forty minutes, and the bath was then tested for acetic acid, the bath was found to contain 25.92% acetic acid and, after four washings with plain water, the particles contained only 0.04% acetic acid. This contrasts with the results obtained when a cellulose acetate dope of the same concentration was extruded into a precipitating liquid of the same composition through an orifice of circular cross-section having an area of 0.0487 sq. in. In the latter case, the precipitating liquid was found to contain only 20.32% acetic acid and, after four washings, the smooth, dense pellets thus obtained still contained 0.11% acetic acid. The thin, substantially flat particles obtained by extruding through the elongated slot are fluffier and of very low density as compared to the pellets obtained using a round extrusion orifice. It required only 25.3 grams of such cellulose acetate particles to fill a 4 ounce container, whereas it required 36.8 grams of cellulose acetate pellets produced by extruding the dope through an orifice of circular cross-section to fill a container of the same capacity.

Some variations and modifications may be made in practicing the invention without departing from the spirit and scope thereof, and the invention is not to be limited therefor except as defined in the appended claims.

I claim:

1. Apparatus for precipitating cellulose acetate in the form of thin, substantially flat porous particles having rough crepe-like surfaces from acid dope formed by acetylation of cellulose comprising, in combination, a nozzle, an elongated extrusion slot having a length of from one-half to one and one-half inches, and a width of from one-eighth to one-fourth inch in the nozzle, a chamber containing a bath of dilute aqueous precipitating liquid, means for supporting the nozzle below the surface of the bath, means for forcing dope through the elongated slot in the nozzle into the liquid, a cutting means rotatably supported adjacent the nozzle and positioned so that when it is rotated the cutting edge thereof strikes against the face of the flat stream of dope extruded through the slot, and means for continuously agitating the precipitating liquid.

2. Method of precipitating cellulose acetate in the form of thin, substantially flat porous particles having rough crepe-like surfaces from acid dope formed by acetylation of cellulose which comprises extruding a stream of the acid dope of substantially flat cross-section below the surface of a bath of dilute aqueous precipitating liquid, and simultaneously contacting the face of the stream of dope with a cutting means to cut it into substantially flat sections and agitating the liquid, said extrusion and cutting rates being in timed relation to produce sections of substantially uniform length, and the agitating rate also being in timed relation to carry away the sections and the acid formed by the precipitation and prevent local concentration of the products of the precipitation.

GEORGE T. DETWILER.

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