Aug. 7, 1962 J. W. SOEHNGEN 3,047,906

WET SPINNING APPARATUS AND START UP PROCESS

Filed Dec. 11, 1959

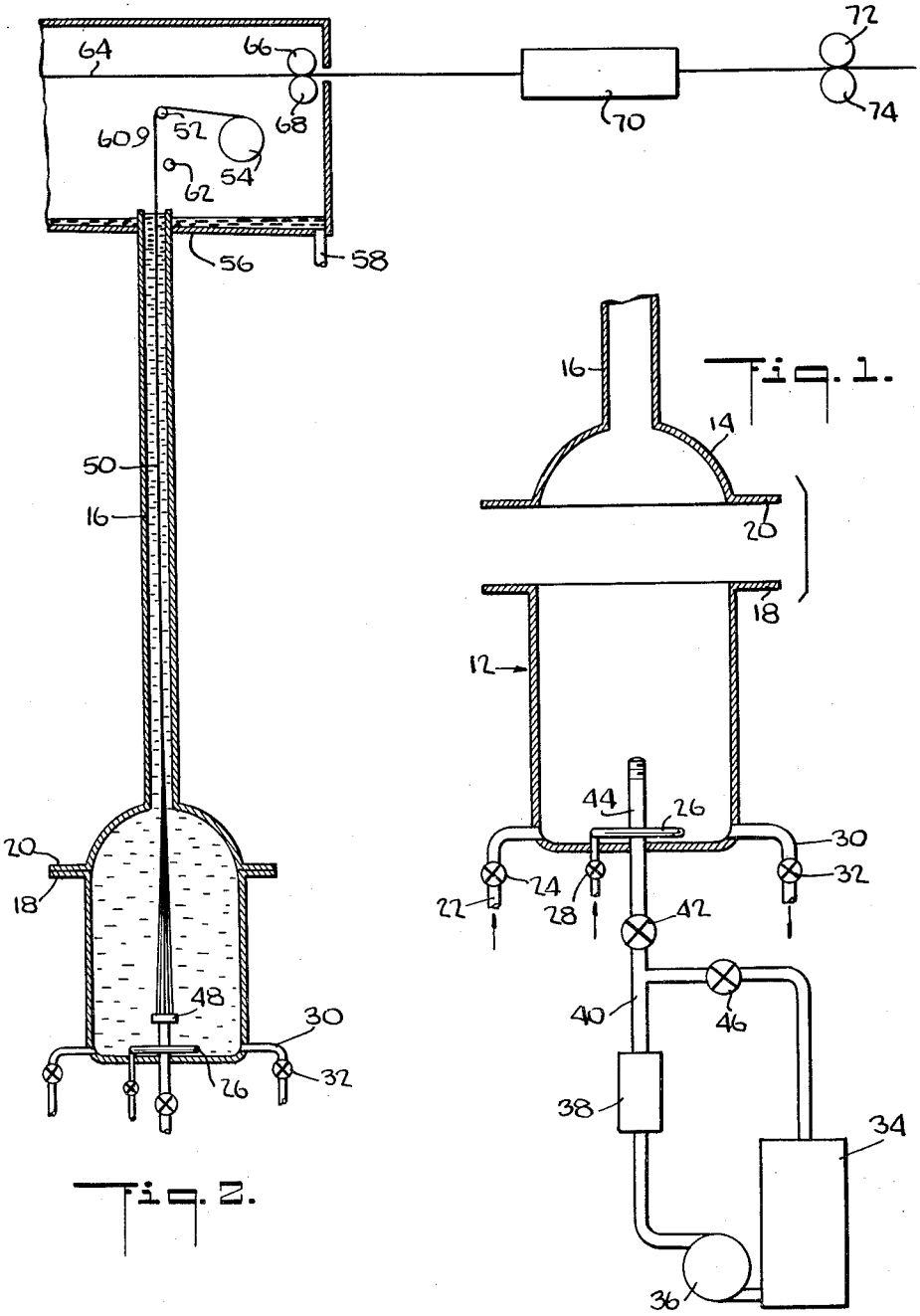


Fig. 1.

Fig. 2.

United States Patent Office

3,047,906
Patented Aug. 7, 1962

3,047,906

WET SPINNING APPARATUS AND START UP PROCESS

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Filed Dec. 11, 1959, Ser. No. 858,944
17 Claims. (Cl. 18—8)

The present invention relates to a novel process for starting up wet spinning.

Wet spinning is one of the basic procedures by which synthetic filamentary materials may be produced. In this procedure a solution of a filament-forming material is extruded continuously through small orifices in a device known as a spinnerette into a liquid which is a non-solvent for the filament-forming material. The filament-forming material precipitates out of solution and is pulled out from the precipitating bath (also called coagulant or spin bath) continuously as filaments.

Once in operation, wet spinning under the proper conditions will proceed smoothly. The act of commencing spinning, however, oftentimes presents a problem due to breakage of the filaments. Where the filaments are to be withdrawn downwardly from the spinnerette, if their density is greater than that of the coagulant they will fall through the coagulant in the same direction that it is desired they move so that they can readily be grabbed, threaded through the necessary post-spinning equipment and the process placed in continuous operation. Where spinning is effected in upward direction obviously gravity will not help in starting operation if the filamenary material has a higher density than the coagulant. The problem is additionally complicated when the filaments are intended to move upwardly through a narrow elongated tube containing coagulant.

These problems are encountered when wet-spinning upwardly in accordance with the disclosure of U.S. application Serial No. 638,414 filed February 5, 1957 in the name of Jesse L. Riley, wherein there is described a process for wet-spinning solutions of organic acid esters of cellulose in solvents comprising halogenated hydrocarbons such as methylene chloride into a spin bath containing a lower aliphatic alcohol as non-solvent for the cellulose ester. While in all processes the freshly formed filaments are fragile, in the Riley process they are specially so since the spin bath exerts an appreciable swelling action on the filaments due to its content of an appreciable amount of solvent for the filamentary material.

It is therefore an object of the present invention to provide a process for starting up a wet-spinning operation.

It is a further object of the invention to provide a process for reliably commencing upward wet-spinning.

Another object of the invention is to provide a process for starting up wet spinning of cellulose ester solutions into spin baths containing appreciable amounts of solvent for the cellulose ester.

Still another object is to provide an apparatus especially suited for starting up and continuing the wet spinning of cellulose triacetate solutions into spin baths containing appreciable amounts of solvent for the cellulose triacetate.

Other objects and advantages will become apparent from the following detailed description and claims, wherein all proportions are by weight unless otherwise specified.

In accordance with one aspect of the invention wet spinning is to be effected into a spin bath exerting a swelling action on the filament-forming material and the wet spinning is initiated into a coagulant which exerts less swelling action on the material than the spin bath to render the filaments tough so that they can withstand

handling and to prevent adhesion of the extruded material to the spinnerette face. One way in which this can be carried out is to employ as the spin bath a mixture of a solvent and a non-solvent for the filament-forming material or a mixture of two liquids of unequal swelling action and to employ as the initial coagulant a medium richer in the non-solvent or poorer swelling liquid than the spin bath. Following the change-over from initial coagulant composition to final spin bath composition and not until spin bath of the final composition surrounds the spinnerette and preferably not until such spin bath is at least 2 inches beyond the spinnerette in the case of co-current flow of dope and spin bath, the draw down ratio (i.e. ratio of linear speed of take-up to linear speed of extrusion) is increased from an initial value of less than about 1 to a value in excess of about 2 and preferably in excess of about 5. If the increase in draw down ratio is effected prematurely the filaments may break, necessitating starting over.

One way in which it can be determined whether the spinnerette is yet surrounded by spin bath of final composition or is still surrounded by initial coagulant is by computation of the time needed, taking into consideration the volumetric capacity of the equipment, the rate of feed and the like. Where possible, preferably it is determined visually as by the location of an interface when the different liquids are immiscible or of markedly different densities. For example, when the initial coagulant is methanol and the final spin bath methylene chloride-methanol and the liquids move upwardly, the heavy final spin bath merely displaces the lighter methanol rather than mixing with it so that an interface is visible even though the liquids would be miscible if stirred.

In accordance with another aspect of the invention when initiating spinning measures are taken to prevent fouling of the spinnerette orifices due to blocking by the filament-forming material upon its extrusion into non-solvent. To this end the spinnerette is covered by solvent, just sufficient dope is extruded through the spinnerette to cover the exit ends of the spinnerette orifices with dope, so as to form a crown of the dope and the solvent is drawn off before dissolving away the crown, leaving the crown to protect the spinnerette orifices. In this manner it is ensured that upon subsequent surrounding of the spinnerette by a non-solvent medium coagulation will occur outside of the spinnerette rather than within it as would occur if the non-solvent could percolate through the spinnerette orifices.

In accordance with still another aspect of the invention, the process is initiated through the dope supply pipe which feeds the spinnerette, except that the spinnerette is absent. The spin pot into which the dope supply pipe extends is filled with solvent above the top of the dope supply pipe to prevent formation of a skin inside the pipe, and a bubble of dope is extruded. The end of the dope supply pipe is wiped clean to remove any skin on the bubble as well as any film which might have been dislodged by the dope in pasisng through the pipe. The spinnerette, suitably dressed with filter cloths, is wet with solvent to remove occluded air and to wet the dressing and is secured to the end of the dope supply pipe to be further treated as described hereinabove. By contrast, if the spinnerette is positioned on the dope supply pipe before wiping away a bubble of dope from the pipe then any film loosened by the flow of dope would clog the inside of the spinnerette.

The novel process is especially useful in combination with the wet-spinning process described in the above-identified Riley application Serial No. 638,414 wherein the solvent of the extruded solution comprises at least about 85% by weight of a halogenated alkane such as methylene

chloride and up to about 15% by weight of one or more alkanols such as methanol, ethanol, isopropanol, propanol, butanol, or the like. Small amounts of water will not interfere.

The coagulant bath may comprise the same classes of liquids as the solution of filmament-forming material except that the concentration of alkanol, which is a non-solvent for organic acid esters of cellulose, is much greater. It has been found that when the concentration of methylene chloride in the alcoholic coagulant bath, C, is related to its temperature in ° C., T, by the equation $C=75¼-T\pm5$ the tenacity and elongation of the resultant filament will both simultaneously be higher than if the concentration is increased or decreased beyond the value according to the equation. The temperature generally ranges from about 15 to 45° C. which means the methylene chloride concentration generally ranges between about 25 and 65%.

The organic acid ester of cellulose filament-forming material can be the acetate, propionate, butyrate, acetate-formate, acetate-propionate, acetate-butyrate, or the like. Preferably it contains fewer than about 0.29 free hydroxyl groups per anhydroglucose unit, e.g. cellulose acetate having an acetyl value of at least about 59% by weight calculated as combined acetic acid and hereinafter referred to as cellulose triacetate.

The invention will be further described with reference to the accompanying drawing wherein:

FIG. 1 is a schematic illustration of a portion of an apparatus for commencing wet spinning of cellulose triacetate from a dope principally comprising methylene chloride into a spin bath containing precipitating proportions of methanol along with methylene chloride; and

FIG. 2 is a schematic illustration of a portion of the apparatus of FIG. 1 plus additional apparatus.

Referring first to FIG. 1, there is shown a spin pot 12 which can be attached to a lid 14 carrying a spinning column 16 by means of flanges 18 and 20 on the pot 12 and the lid 14, respectively. A liquid inlet 22 provided with a valve 24 controls admission of liquid to the spin pot 12. An annular sparger 26 with a valve 28 controls admission of spin bath to the spin pot. If desired, inlet 22 could communicate with the sparger 26 either inside or outside the spin pot with no change in operation. A liquid outlet 30 with valve 32 controls drainage of liquid from the spin pot.

A dope of 21% cellulose triacetate in 90/10 methylene chloride/methanol is held in a reservoir 34 from which it is pumped by a pump 36 through a candle filter 38 into a T-pipe 40, one outlet of which, through a valve 42, communicates with a dope supply pipe 44 and the other outlet of which communicates through a valve 46 with a return to reservoir 34. At least one of valves 42 and 46 is always open so that dope is constantly flowing. Control means (not shown) may be provided to prevent both valves 42 and 46 from being simultaneously closed.

Before commencing spinning valve 46 is open and valve 42 is closed. Valve 32 is also closed. To commence spinning methylene chloride is supplied to fill the spin pot 12. Valve 42 is cracked, dope is allowed to form a bubble at the outlet of pipe 44 and valve 42 is closed. The bubble is wiped away along with any film or skin. A spinnerette 48 (FIG. 2) is screwed onto the end of pipe 44 and the methylene chloride level is brought to about ⅛ inch above the top of the spinnerette. Just enough dope is supplied through pipe 44 (by operation of valve 42) to form a bubble or crown on the spinnerette 48 due to contact of the dope with the atmosphere, the crown commencing below the liquid level in the pot 12 but projecting slightly above said level at its high point. Valve 32 is opened, draining away the methylene chloride and leaving the crown of dope which prevents solidification of filament-forming material in the spinnerette openings due to contact with the atmosphere or with the non-solvent which is subsequently supplied.

Methanol, or methylene chloride-methanol at least about 20% richer in methanol than the final spin bath, is then supplied through 22 above the top of the dope crown and is continued to be supplied with valve 32 open to maintain its level constant. Valve 42 is opened and dope is fed at operating speed. The crown of dope, which now is a coagulated swollen body of cellulose triacetate, is pulled away from the spinnerette, forming filaments under the drafting action. The filament bundle or tow 50 is continuously pulled away until all holes are spinning freely. The tow of filaments 50 is then cut to discard the first portion and crown, and that portion which is "connected" to the spinnerette is clamped on a string which is inserted down through the column 16. The string is drawn up through the column and the tow is passed over a pin 52 and is thrown onto a driven waste roll 54 (FIG. 2) which takes up the tow at a draw down ratio of about 1 or less.

Lid 14 together with column 16 is then lowered, column 16 sliding through an opening in headbox 56 (FIG. 2), and flanges 18 and 20 are locked to one another. Valve 32 is closed so that methanol starts filling the column 16. When its level is about 6 to 60 inches above the spinnerette 48, e.g. 40 inches, its flow rate is cut to nothing gradually over about 1 minute while, simultaneously over substantially the same time, feeding of spin bath through sparger 26 is commenced and brought to operational flow rate, e.g. about 20 liters per minute. Alternatively, the methanol flow rate can be decreased and pure methylene chloride fed through sparger 26 to arrive at the proper final concentration; the former procedure is preferred, however, since it ensures that the methanol and methylene chloride are well mixed and requires the least amount of pure single solvents. All the liquids withdrawn through 30 until this stage are segregated for separate recovery since their compositions differ from that of the spent spin bath recovered during normal operation and since they may contain a relatively high proportion of filament-forming material which would necessitate special recovery procedures to prevent clogging.

Spin bath rises in column 16 and overflows into headbox 56 which is inclined toward drain 58 through which spent spin bath is withdrawn for regeneration. When the spinnerette is surrounded by spin bath, rather than by methanol, and the demarcation between the spin bath and methanol is at least 2 inches beyond the spinnerette the speed of waste roll 54 is increased to the desired take-up speed. Pin 60 is moved to the right and pin 62 to the left (relative to their illustrated positions) to adjust the tension on tow 50.

The tow 50 is then severed between pin 52 and roll 54 and is thrown onto a master tow 64 made up of several tows from adjacent columns. The master tow 64 passes between driven squeeze rolls 66, 68 which express entrained spent spin bath, the spin bath falling to the bottom of head box 56 and being withdrawn at 58. The tow 64 leaves headbox 56 through an opening in the end wall, is dried in drier 70, is pulled at predetermined speed by rolls 72, 74, and is then processed in conventional manner, e.g. is lubricated and taken up as continuous filament yarn or cut into staple fiber. While the invention has been described with reference to a master tow 64 obviously the tow 50 from each spinning column can be separately collected in which case starting up will necessitate threading of the tow, cut between pin 52 and waste roll 54, between the squeeze rolls 66, 68 and the subsequent appaartus.

It is to be understood that the foregoing detailed description is merely given by way of illustration and that many variations may be made therein without departing from the spirit of my invention.

Having described my invention, what I desire to secure by Letters Patent is:

1. In the wet spinning of a dope of filament-forming material through a spinnerette into a spin bath exerting a swelling action on said material, the improvement

which comprises commencing said wet spinning into a coagulant of lesser swelling action than said spin bath and withdrawing the formed filaments at a speed below the desired spinning speed, changing the composition of the coagulating medium from that of said coagulant to that of said spin bath, and increasing the withdrawal speed to the desired spinning speed when said spinnerette is surrounded by said spin bath.

2. The process set forth in claim 1, wherein said filament-forming material comprises cellulose triacetate.

3. The process set forth in claim 1, wherein the draw down ratio when commencing wet spinning into said coagulant of lesser swelling action is at most about 1 and is increased to at least about 2 when said spinnerette is surrounded by said spin bath.

4. In the wet spinning of a dope of filament-forming material through the orifices of a spinnerette into a spin bath, the improvement which comprises surrounding said spinnerette with a solvent for said filament-forming material, extruding sufficient dope through said spinnerette to form a crown of dope on said spinnerette, withdrawing said solvent, surrounding said spinnerette with a coagulating medium for said filamentary material, and thereafter continuously extruding said dope through said spinnerette.

5. The process set forth in claim 4, wherein said spin bath exerts a swelling action on said filament-forming material, said coagulating medium initially having a composition which exerts a lesser swelling action on said material and its composition being changed to that of said spin bath in the course of spinning.

6. In the wet spinning of a dope of filament-forming material through a spinnerette into a spin bath comprising a mixture of a solvent and a non-solvent for said filament-forming material, the improvement which comprises commencing said wet spinning into a medium at least about 20% by weight richer in non-solvent than said spin bath and withdrawing the formed filaments at a draw down ratio of at most about 1, changing the composition from that of said medium to that of said spin bath, and increasing the draw down ratio to at least about 2 when the spinnerette is surrounded by said spin bath.

7. The process set forth in claim 6, wherein the spin bath and filaments move cocurrently and the increase in draw down ratio is effected after spin bath of the final composition is at least 2 inches beyond the spinnerette.

8. The process set forth in claim 7, wherein the draw down ratio is increased to at least 5.

9. The process set forth in claim 6, wherein said filament-forming material comprises an organic acid ester of cellulose.

10. The process set forth in claim 6, wherein said dope comprises an organic acid ester of cellulose having fewer than about 0.29 free hydroxyl group per anhydroglucose unit dissolved in a halogenated hydrocarbon and said spin bath comprises a halogenated hydrocarbon and sufficient lower aliphatic alcohol to precipitate the cellulose ester.

11. The process set forth in claim 6, wherein said dope comprises an organic acid ester of cellulose having fewer than about 0.29 free hydroxyl group per anhydroglucose unit dissolved in a halogenated lower alkane and said spin bath comprises a halogenated lower alkane and sufficient lower alkanol to precipitate the cellulose ester.

12. The process for commencing wet spinning of a dope of filament-forming material in a solvent through a spinnerette into a spin pot containing a spin bath comprising a mixture of said solvent and a non-solvent, which comprises circulating said dope in a closed system, supplying said solvent to said spin pot to a level above said spinnerette, feeding dope from said system to form a crown on said spinnerette, draining away said solvent from said spin pot, continuously supplying to said spin pot a liquid richer in said non-solvent than said spin bath and withdrawing it therefrom at a rate sufficient to maintain its level above said spinnerette, pulling said crown and filaments connected thereto away from said spinnerette and withdrawing said filaments at a draw down ratio of at most about 1, changing the composition of coagulant supplied to said spin pot from that of said liquid to that of said spin bath, and increasing the draw down ratio to at least about 2 when the spinnerette is surrounded by said spin bath.

13. The process for commencing wet spinning of a dope of cellulose triacetate in a solvent comprising at least about 85% by weight of methylene chloride through a spinnerette into a spin pot containing a spin bath comprising about 25 to 65% of methanol and about 75 to 35% of methylene chloride, which comprises circulating said dope in a closed system, supplying methylene chloride to said spin pot to a level above said spinnerette, feeding dope from said system to form a crown on said spinnerette, draining away the methylene chloride from said spin pot, continuously supplying to said spin pot a liquid at least about 20% richer in methanol than said spin bath and withdrawing it therefrom at a rate sufficient to maintain its level above said spinnerete, pulling said crown and filaments connected thereto away from said spinnerette and withdrawing said filaments at a draw down ratio of at most about 1, changing the composition of coagulant supplied to said spin pot from that of said liquid to that of said spin bath, and increasing the draw down ratio to at least about 2 when the spinnerette is surrounded by said spin bath.

14. The process for commencing wet spinning of a dope of filament-forming material in a solvent through a dope supply pipe surmounted by a spinnerette into a spin pot containing a spin bath comprising a mixture of said solvent and a non-solvent which comprises circulating said dope in a closed system, supplying said solvent to said spin pot to a level above said dope supply pipe with said spinnerette removed from the latter, feeding dope from said system to form a bubble atop said dope supply pipe, wiping away said bubble, positioning said spinnerette on said dope supply pipe, bringing the level of said solvent slightly above said spinnerette, feeding additional dope to form a crown on said spinnerette, draining away the solvent from said spin pot, continuously supplying to said spin pot a liquid richer in said non-solvent than said coagulant and withdrawing it therefrom at a rate sufficient to maintain its level above said spinnerette, pulling said crown and filaments connected thereto away from said spinnerette and withdrawing said filaments at a draw down ratio of at most about 1, changing the composition of coagulant supplied to said spin pot from that of said liquid to that of said spin bath, and increasing the draw down ratio to at least about 2 when the spinnerette is surrounded by said spin bath.

15. The process for commecing upward wet spinning of a dope of cellulose triacetate in a solvent comprising at least about 85% by weight of methylene chloride through a dope supply pipe surmounted by a spinnerette into a spin pot containing a spin bath comprising about 25 to 65% of methanol and about 75 to 35% of methylene chloride, which comprises circulating said dope in a closed system, supplying methylene chloride to said spin pot to a level above said dope supply pipe with said spinnerette removed from the latter, feeding dope from said system to form a bubble atop said dope supply pipe, wiping away said bubble, positioning said spinnerete on said dope supply pipe, bringing the level of methylene chloride slightly above said spinnerette, feeding additional dope to form a crown on said spinnerette, draining away the methylene chloride from said spin pot, continuously supplying methanol to said spin pot and withdrawing it therefrom at a rate sufficient to maintain a methanol level above said spinnerette, pulling said crown and filaments connected thereto away from said spinnerette and withdrawing filaments at a draw down ratio of at most about 1, replacing the feed of methanol to said spin pot by the feed of spin bath, and increasing the draw down ratio to

at least about 2 when the spin bath level is at least 2 inches beyond said spinnerette.

16. A wet spinning apparatus comprising a dope reservoir, a pipe system communicating at two locations with said reservoir, means for circulating dope in said system and reservoir, a spin pot, a dope supply pipe communicating with said system and the interior of said spin pot, first and second valves in said dope supply pipe and in said system respectively whereby dope from said system can be discharged into said spin pot or can be recirculated, a liquid inlet communicating with said spin pot for supplying starting up liquid, a liquid outlet for removing starting up liquid from said spin pot, and a spin bath inlet communicating with said spin pot.

17. A wet spinning apparatus comprising a spin pot, a spinning column communicating with said pot, a dope reservoir, a dope supply pipe communicating with said reservoir and discharging upwardly into said spin pot, a first valve in said dope supply pipe, a return pipe communicating with said reservoir and with said dope supply pipe between said first valve and said reservoir, a second valve in said reservoir, a pump for causing flow of dope through at least one of said valves, an inlet for supplying starting up liquid to said spin pot, an outlet adjacent the bottom of said spin pot for draining starting up liquid from said spin pot, a spin bath inlet communicating with said spin pot, means for catching overflowing spent spin bath from said column, and means for taking up filamentary material after its passage through said column.

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