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SOLUTION SPINNING OF POLYPROPYLENE

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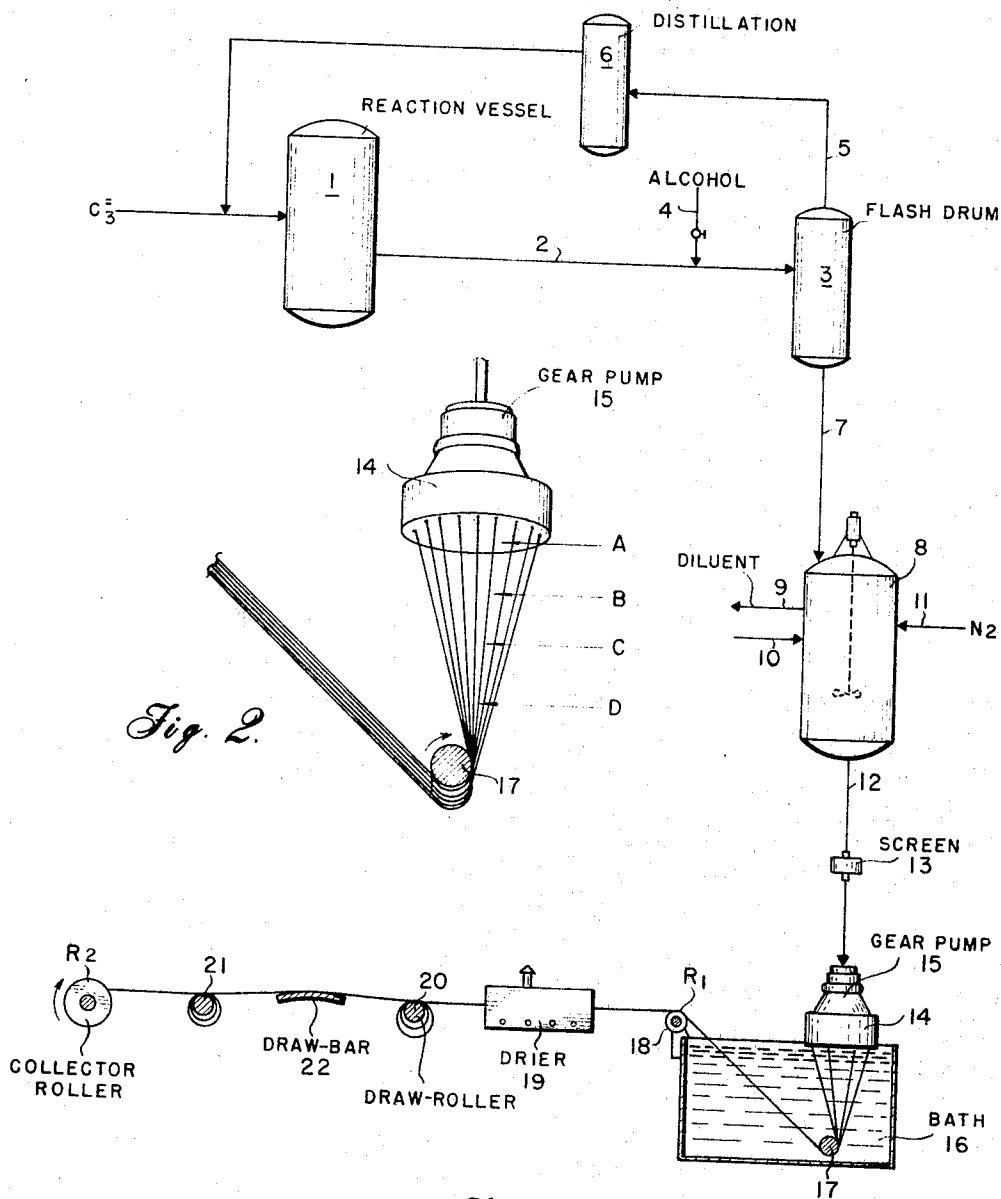


Fig. 1.

Fig. 2.

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SOLUTION SPINNING OF POLYPROPYLENE

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7 Claims

ABSTRACT OF THE DISCLOSURE

Polypropylene is spun from a concentrated solution in a solution spinning process through a short air gap into a water bath. The fibers are dried in a heated oven and may be drawn in line to produce high strength fibers.

BACKGROUND OF THE INVENTION

The present invention is directed to a process for spinning a solution of polypropylene to produce fibers of high strength. More specifically, the invention is a solution spinning process comprising spinning a concentrated polypropylene solution through a short air gap into a water bath, drying the fibers in a heated oven, and thereafter drawing the fibers to produce fibers of high strength.

There are three basic types of known spinning processes for the production of synthetic fibers or filaments and they are: (1) melt spinning, (2) dry spinning, and (3) wet spinning. Of the three, melt spinning is used in the commercial production of polypropylene fibers.

In the melt spinning of polypropylene, polypropylene in the form of powder or pellets is introduced to a melt spinning device wherein the polypropylene is melted and extruded through a spinnerette in the shape of fibers. The fibers are cooled by air and are thereafter drawn and otherwise treated. The melt spinning process allows fairly high spinning speed, but requires considerable extrusion pressure and high melt temperatures.

The dry spinning technique involves the extrusion of a solvent solution of polymer through a spinnerette into a stream of heated gas to rapidly evaporate the solvent from the polymer and thus form the polymer filaments. The spinning of polypropylene by a conventional dry spinning process wherein a polypropylene solution is spun and the fibers passed through an evaporation tube is described in U.S. 3,032,384. Another dry spinning process is described in U.S. 3,161,709.

In wet spinning, a polymer solution is extruded through a spinnerette directly into a coagulating bath. The function of the bath is to remove the proper amount of solvent from the fiber or filament. In the wet spinning of polypropylene, there has been disclosed a process using a fatty acid solvent when spinning into a water bath containing a base as in U.S. 3,017,238. Another process is described in U.S. 3,048,465 for the wet spinning of polypropylene. However, the spinning speeds are very slow and several extraction baths are suggested.

None of the foregoing patents describe or suggest a process for solution spinning solutions of polypropylene in acceptable reaction solvents to produce fibers of high strengths at commercially feasible rates. In the process of the present invention, a concentrated solution of polypropylene is spun through a short air gap into a water bath.

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SUMMARY OF THE INVENTION

The present invention may be briefly described as a solution spinning process which comprises forcing a concentrated solution of polypropylene through a spinnerette and passing the fibers through a short air gap into a water bath, drying the fibers preferably in a heated oven to remove the solvent, and then stretching or drawing the fibers. It is preferred that the entire operation be in line, although the spinning, drying and drawing may be separate operations.

According to the present invention, it has been found that a concentrated solution of polypropylene having a concentration of between 35 and 55 percent may be solution spun from a solvent selected from the C₉ to C₁₁ paraffins or from xylene, all of which solvents may be used as reaction diluents. The preferred solvent is n-decane (C₁₀). It is preferred that the concentration of the solution be as high as possible to obtain good fiber quality and this is obtained by operating at about the boiling point of the particular solvent. It has been found that to maintain high rates of speed that the air gap between the die head of the spinnerette and the water bath must have a small air gap of between 1/16 to 1 1/2 inches. The fibers are quenched in the water bath and collected and thereafter heated in a heated oven or by heating elements such as infrared heaters or the like to remove the solvent and thereafter are drawn to produce the fibers of high strength.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 discloses a schematic diagram of the solution spinning process of the present invention.

FIG. 2 illustrates the advantage of a short air gap in a solution spinning process.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to FIG. 1 which illustrates a preferred embodiment of the present invention, propylene is polymerized in a reaction vessel 1. To the reaction vessel 1 is added a diluent selected from the C₉ to C₁₁ paraffins or xylene, propylene, and a Ziegler-type catalyst preferably a co-crystallized aluminum titanium chloride together with an aluminum alkyl such as aluminum triethyl. The reaction mixture is removed from the reaction vessel 1 by line 2 and is passed to a flash vessel 3. An alcohol such as methanol may be added in line 2 by line 4 to stop the reaction. In the flash vessel 3 the unreacted monomer and methanol is flashed overhead by line 5 where it is separated such as in a distillation column 6 to be recycled. The polypropylene solution is removed from flash vessel 3 by line 7 and passed to a solution vessel 8 wherein the solution is concentrated to about 45 percent polypropylene by weight by removing diluent by line 9 or addition of diluent by line 10 as needed. The concentrated polypropylene solution may be forced out of solution vessel 8 by an inert gas such as nitrogen introduced by line 11 from the solution vessel 8 to line 12 whereby it passes through a screen 13 to remove any particles by passing to the spinnerette 14. To maintain a continuous and constant solution flow, a gear pump 15 may be used in line 12 to force the polypropylene solution to spinnerette 14.

The spinnerette 14 has a die head having a die with a plurality of holes which may have a diameter of about 0.006 to 0.030 inch. The holes are arranged in a suitable pattern such as in a series of concentric circles or in parallel lines. A suitable die for the spinning of polypropylene was one having 36 holes each of which had a diameter of 0.016 inch and the holes arranged in a circle.

The concentrated solution is forced through spinnerette 14 into the die head at temperatures ranging from 30° F. below the boiling point up to the boiling point of the solvent. Since the specific solvent which is chosen, the concentration of the solution being spun and the molecular weight of the polypropylene all influence to some degree the viscosity of the concentrated solution of polypropylene, the temperature may be selected within a relatively wide range to adjust the spinning conditions so as to obtain fibers of good quality.

The die head of the spinnerette is positioned above a water bath 16 with an air gap or space of between 1/16 and 1/2 inches and preferably the range is between 1/4 and 1/2 inch. The advantage of a short air gap in solution spinning is illustrated in FIG. 2 wherein it is shown that the distance between each strand of fiber as the strands are spun from spinnerette 14 into water bath 16 are greatest at point A as compared to B, C, and D thus preventing entanglement as the strands are collected on guide roll 17 in the water bath. The guide roll 17 may be short or long depending on the configuration of holes in the die head of the spinnerette. In any event, the short air gap prevents strand entanglement and subsequent breakage by the strands coming into contact one with another before being cooled and quenched by the water bath 16. After the fibers pass through the water bath 16, they may be collected on a roll R₁ or may be passed over guide roll 18 directly to an oven 19. The fibers are heated to remove the solvent in oven 19. The fibers are then drawn by use of draw rolls 20 and 21 over a heated draw bar 22 at draw ratios between 2:1 to 6:1 to form fibers of high strength. Preferably the draw ratio is about 4:1 to obtain polypropylene fibers of suitable tenacities. The fibers are then collected on a roll R₂.

While a continuous in-line process has been described, it is understood that polypropylene may be reacted under conditions different from those described above and may be merely added to concentration vessel 8 for spinning by the preferred process. Likewise the fibers which have been spun may be collected on roll R₁ to be air dried and may thereafter be drawn using a device such as the draw rolls 20 and 21 with the fiber drawn over a heated draw bar 22 at draw ratios of 2:1 to 6:1. Fibers which are air dried may eliminate the need of drying oven 19 or it may be used in addition to the air drying. Other drying techniques and equipment than an oven 19 may be used to remove the solvent from the fibers.

The present invention will be further described by the following examples which are given by way of illustrations and not as limitations on the scope of the invention.

EXAMPLE 1

To a reaction vessel is added 1,000 grams of polymerization grade decane and a catalyst consisting of co-crystallized aluminum reduced titanium trichloride and aluminum triethyl. The temperature of the mixture is maintained at 150° F. and propylene gas is added continuously at a pressure of 200 p.s.i.g. until approximately 400 grams have been polymerized. The mixture is then passed to a solution vessel where a small amount of alcohol is added to deactivate the catalyst. The temperature is increased to slightly above the boiling point of decane in order to flash off unreacted propylene, alcohol, and an amount of solvent necessary to adjust the solution concentration to 45 percent polypropylene. The solution is thoroughly mixed and the temperature adjusted to 340° F.

The concentrated polypropylene solution is pumped to a spinnerette wherein it is forced through a die having

36 holes each having a diameter of 16 mils. The strands are then passed into a water bath and cooled and quenched. The distance between the die of the spinnerette and the water bath is 1/4 inch and the die bath is maintained at room temperature.

By the foregoing example, a continuous and integral process from the polymerization of polypropylene to the production of a fiber is illustrated.

EXAMPLES 2-6

Illustrated in the following table, Table I, are several runs wherein polypropylene in various solvents and concentrations were passed through a spinnerette containing 36 holes, each hole being 16 mils in diameter under the conditions shown in the table, and into a water bath at room temperature. In the operation of the various runs, it was observed that strand separation was maintained at air gaps of about 1/4 to 1/2 inch whereas strand entanglement occurred if the air gap between the die head of the spinnerette and the water bath was about 2 to 3 inches. As an aid to strand separation, a wetting agent could be added to the water bath such wetting agents being normal detergents or surface active agents such as Triton X-100. The ability to spin at the rates shown with proper strand separation and ease of take up illustrate the advantages of the process of the present invention.

TABLE I

	Example				
	2	3	4	5	6
Polymer.....	L-4295	L-4295	F-148	B-3645	L-4295
Solvent.....	C ¹⁰	C ⁹	Xylene	C ¹⁰	C ¹⁰
Concentration, percent.....	50	50	40	50	50
Solution Temperature ° F.....	342	295	260	315	340
Pump rate, cc./min.....	5.9	5.9	13.4	5.9	11.8
Air gap, minute.....	1/4	1/4	1/2	1/4	1/4
Take-up speed, f.p.m.....	200	200	990	200	200
Drying.....	(1)	(2)	(2)	(2)	(2)
Draw device.....	(3)	(3)	(4)	(3)	(3)
Draw ratio.....	4.1:1	4:1	2:1	4:1	4:1
Fiber size, denier.....	2.7	2.7	1.8	2.5	5.0
Tenacity, gm./den.....	5.5	4.8	3.3	3.9	4.1

¹ Vacuum oven.

² Room condition.

³ Oven.

⁴ 250° F. Draw pin.

It is noted that the fibers formed all have a tenacity above 3 and several above 4 which would mean that the fibers could be used in most fiber applications. For example, textile fibers in carpet applications would have tenacities about 2 to 3.

The nature and objects of the present invention having been completely described and illustrated and the best mode thereof set forth, what I wish to claim as new and useful and secure by Letters Patent is:

1. A solution spinning process which comprises forcing a concentrated spinning solution of polypropylene in a solvent selected from the group consisting of the C₉ to C₁₁ paraffins and xylene, said solution having a concentration of between 35 to 55% by weight polypropylene, through a spinnerette and passing the spun polypropylene fibers into a water bath through an air gap of between 1/16 and 1 1/2 inches.

2. A process according to claim 1 wherein said solvent is n-decane.

3. A process according to claim 1 wherein said air gap is between 1/4 and 1/2 inch.

4. A process according to claim 1 wherein said concentrated spinning solution is a reaction mixture concentrated to between 35 and 55 percent by weight polypropylene.

5. A process according to claim 1 wherein said polypropylene fibers are removed from said water bath, drying said fibers to remove the solvent and drawing said fibers.

6. A process according to claim 5 wherein said polypropylene fibers are drawn at draw ratios between 2:1 to 6:1.

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7. A process according to claim 5 wherein said polypropylene fibers are air dried before drawing.

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