

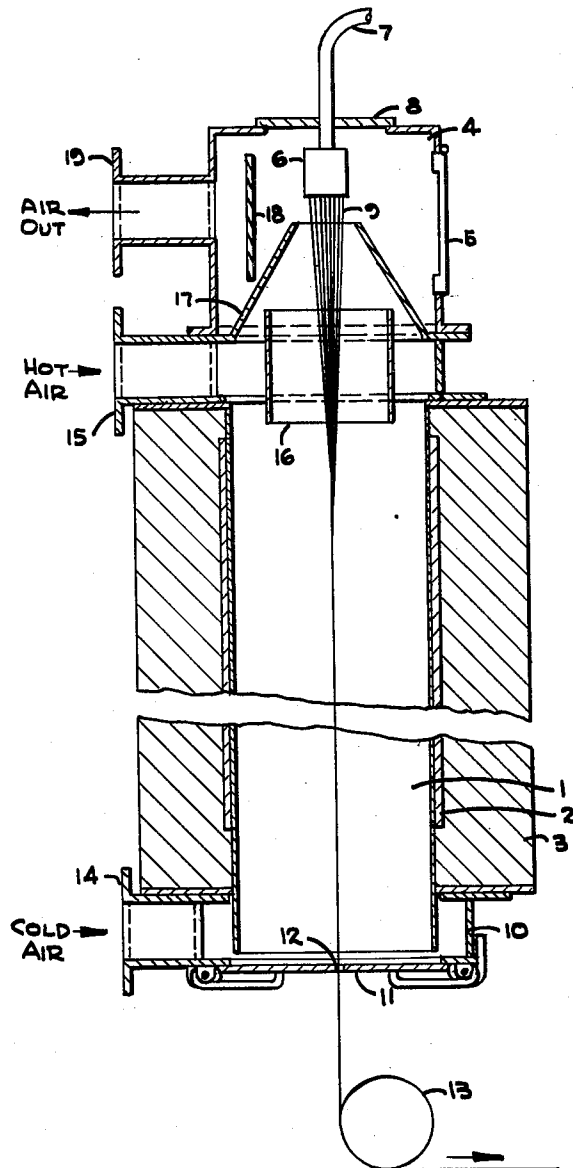
April 28, 1953

W. H. HAMMOND ET AL

2,636,217

DRY SPINNING APPARATUS

Filed July 20, 1949



LAURENCE C. HOLT
ROGER L. SCHAEFER
WALTER H. HAMMOND
INVENTOR.

BY *Donald Haeffele*
ATTORNEY

UNITED STATES PATENT OFFICE

2,636,217

DRY SPINNING APPARATUS

Walter H. Hammond, Miamisburg, and Laurence C. Holt, Dayton, Ohio, and Rodger L. Schaefer, Decatur, Ala., assignors, by mesne assignments, to The Chemstrand Corporation, a corporation of Delaware

Application July 20, 1949, Serial No. 105,790

6 Claims. (Cl. 18-8)

1

This invention relates to an apparatus and procedure for preparing synthetic resinous structures. More specifically the invention relates to an improved method of spinning fibers by extruding solutions of synthetic resins into a coagulative medium.

The preparation of linear structures, such as films, fibers, rods, and other shapes, by forcing solutions of resins through a suitable die into a gaseous medium which removes the solvent and precipitates the resin in a continuous form, is well known. Many methods and types of apparatus for practicing the dry spinning technique have been described in the published literature. These publications show various methods of circulating the coagulation medium and structures for manipulation of the extruded fiber. In general there are two methods described for the movement of the gaseous medium with respect to the fiber, countercurrent and concurrent, each of which has inherent undesirable attributes as well as beneficial aspects. The countercurrent flow of the spinning medium is effective in reducing the residual solvent in the fiber to a minimum, because the solvent free gas is in contact with the fiber nearly exhausted of solvent. This method, however, provides the initial coagulation in the presence of gas containing substantial solvent content which retards the evaporation of solvent from the freshly spun fiber. The concurrent flow of the gaseous spinning medium provides a solvent free gas at the spinneret to accelerate the rate of coagulation, but the solvent laden gas in contact with the fiber at the exit end prevents or retards the adequate removal of solvent from the fiber. In each of these conventional procedures one object is achieved at the expense of other desirable objectives.

The primary purpose of the present invention is to provide a method of obtaining rapid coagulation and simultaneously highly efficient solvent removal. A further objective is to provide a novel and useful dry-spinning method by which fibers may be spun effectively and economically.

It has been found that if a dry-spinning operation, conducted in a vertical tower with a downwardly moving fiber, is modified by introducing the solvent free gaseous medium at a point immediately below the spinneret at a temperature substantially that of the boiling point of the solvent, withdrawing the solvent laden gas above the spinneret, and contacting the fiber during its downward course in the tower with a stream of gas at a substantially lower temperature, the

2

beneficial effects of both countercurrent and concurrent fiber spinning may be achieved. By this practice a small amount of the relatively cold gaseous medium is introduced at the bottom of the tower, said amount being from two to 20 percent of the volume of the effluent gas. The relatively cold gas stream passes upward and is heated to a moderately high temperature by the heating elements in the tower walls. At the top of the tower the small amount of gas containing the solvent removed from the fiber is diluted with a large volume of hot gas which provides an atmosphere relatively free of solvent and at a temperature substantially that of the boiling point of the solvent.

The new apparatus and spinning method may be effectively used in the preparation of fibers of the acrylonitrile polymers, such as polyacrylonitrile and copolymers of over 80 percent acrylonitrile and up to 20 percent of other copolymerizable olefinic monomers, for example vinyl acetate, vinyl pyridine, methacrylonitrile, vinylidene chloride, and the esters of acrylic and methacrylic acids.

Conventional dry-spinning practice, involving the use of high boiling solvents, for example N,N-dimethylformamide, ethylene carbonate, N,N-dimethylacetamide and butyrolactone, is often harassed with the development of a yellow or brown discoloration in the fiber. The difficulty is believed to be due to the high spinning temperatures required to effectively remove the solvent. Through the use of the novel method and apparatus described herein fibers free of discoloration may be prepared. The use of high boiling solvents is usually necessary in the preparation of fibers of polyacrylonitrile and copolymers of substantial proportions of acrylonitrile and minor proportions of other monomers, because of the inherent difficulties of preparing suitable spinning solutions. Accordingly, a further purpose of this invention is to provide an effective method of eliminating the color in acrylonitrile fibers.

The essential features of the new method are the extrusion of the resin solution into a countercurrently flowing stream of gas at a high temperature and with a very low solvent vapor content. Shortly after spinning the fiber passes into a zone of countercurrent flow at a lower temperature and usually lower velocity. This lower temperature zone is fed with fresh solvent free air at a point from which the finished fiber is withdrawn. The stream of cold air, as it passes countercurrently with respect to the fiber, car-

3

ries along with it the solvent which has evaporated from the surface of the fiber and is gradually increased in temperature by the heating means provided in that zone. As the stream of air approaches the spinneret it is mixed with a substantially larger stream of solvent free gas at a much higher temperature and the increased volume of hotter gas passes rapidly over the freshly extruded fiber in the vicinity of the spinneret, past the spinneret and is exhausted through a suitable solvent recovery apparatus. The gaseous medium may be moved through the tower by the positive pressure of the hot and cold gas streams entering the apparatus, or it may be withdrawn by means of a suitable exhaust fan.

To be effective the new method should involve the use of suitable baffling means to prevent fiber injury by the force of the gas streams within the tower. The substantial velocity of the hot gas stream entering near the spinneret may damage or even break the freshly spun fiber. Accordingly, the fiber is preferably inclosed in a sheath which deflects the gas stream and directs it in the desired countercurrent direction in close proximity to the freshly spun fiber. If desired the baffle may be tapered in the direction of the spinneret so as to insure the maximum contact of the low solvent containing gas with the freshly spun fiber at the spinneret. It is often desirable to have a deflecting means over the gas exit to prevent a non-uniform gas withdrawal and to assure a more nearly uniform distribution of gas flow around the periphery of the spinneret.

The new method is conducted in a spinning tower comprised of two parts, the tower proper in which the formed fiber is contacted with a countercurrent gas stream at a moderate temperature, and the spinning head or operating zone in which the bulk of the air is introduced at a higher temperature and in which the fiber is actually spun in contact with the stream of gas at the higher temperature. The tower proper is provided with suitable heating elements which may be electrically heated coils of a steam heated means for maintaining the desired temperature at which the solvent has a substantial vapor pressure. For reasons of economy the tower is preferably coated with a substantial layer of a suitable insulation material.

The operating head of the tower is provided with gas inlet and outlet ports, a suitable aperture at the top for the introduction of the spinneret, baffles of suitable size and shape to prevent damage to the fiber and to insure intimate contact of the fiber with the gas stream and a suitable port or window through which the operator may inspect the operation, and if necessary, to manipulate the fiber in initiating the operation or for cleaning the spinneret.

The attached drawing is the cross-sectional view of a spinning tower assembly in which the spinning tower 1 is an elongated tube of any desired cross-section which is heated by a suitable heating element 2 and enclosed by a suitable insulation material 3. The spinning head or operating area 4 is superposed on the tower and is provided with a hinged door 5 for observation and manipulation of the spinneret during the spinning operation. The spinneret 6 is provided with resin solution through conduit 7 passing through the cover plate 8 at the top of the tower. The extruded fiber 9 com-

4

prised of numerous minute filaments passes downwardly through the tower and out at the bottom through an enclosure 10 which is provided with hinged doors 11 having an aperture 12 for the normal passage of the fiber, which leaves the tower and is conveyed to subsequent operations by means of thread guide 13.

A small amount of the necessary gaseous atmosphere is introduced at room temperature through port 14 and is passed up the tower countercurrently with respect to the fiber direction. As it passes into the spinning head or operating zone it is mixed with a larger stream of air at a much higher temperature which enters through port 15. The large volume of hot gas entering the tower is deflected by a cylindrical baffle 16 which directs the gas parallel to the fiber and into the funnel shaped baffle 17 which directs the gas stream in close proximity to the freshly spun fiber at the spinneret. A suitable baffle 18 provides for a uniform withdrawal of gas from the spinning head from which it passes out through passage 19 to a suitable solvent recovery system not shown.

The size and shape of the parts of the tower are not critical. The tower may be any convenient width, but preferably from four to twelve inches, so as to provide an adequate volume of gas in lamellar flow for the removal of the normal solvent content of the fiber without requiring an excessive velocity, and avoiding turbulence effects. The length of the tower will depend upon the desired extent of solvent removal. If complete, or substantially complete solvent removal is desired a tower of ten to 100 times the length of the operating head may be required. The length of the operating head, or the distance between the hot air inlet and the spinneret will also depend upon the size of the fiber, the number of filaments, the volume of hot gas passed into the tower, the temperature of the gas, the type of resin and solvent, the sensitivity of the fiber with respect to discoloration, and the spinning speed. The manipulation of the size and shape of the spinning head to produce the optimum fiber may be practiced by one skilled in the art. In general, the tower length may be from five to 20 times the length of the spinning head, and if solvent recovery is not a serious problem it may be as short as two times the length of the spinning head.

The new method and apparatus are particularly useful in spinning polyacrylonitrile and copolymers of over 80 percent acrylonitrile and up to 20 percent of other polymerizable monomers, such as vinyl acetate and other vinyl esters of carboxylic acids, methyl acrylate and other alkyl esters of acrylic acid, especially those wherein the alkyl group has up to six carbon atoms, methyl methacrylate and other alkyl methacrylates wherein the alkyl group has up to six carbon atoms, styrene, and other vinyl substituted aromatic hydrocarbons, methacrylonitrile and vinyl pyridine. In the preparation of fibers from the acrylonitrile polymers the spinning method utilizes solutions of the desired resin in a solvent, such as dimethylformamide, butyrolactone or ethylene carbonate. Under such circumstances the temperature at the spinneret will be between 140 and 160° C., the countercurrent flow through the tower is initiated with air at room temperature and is gradually heated up to 80 to 100° C. by means of the heating elements in the tower. At the

5

top of the tower the countercurrent stream of air is diluted with a much larger volume of a hot gas, for example at a temperature of 150 to 170° C. By the use of this spinning method acrylonitrile fibers of excellent color may be spun efficiently.

The invention is described by the following claims.

1. A method of preparing fiber, which comprises extruding a solution of a polymer of at least 80 per cent acrylonitrile in a volatile solvent into a countercurrent gas stream, which stream is solvent free gas at room temperature in contact with the finished fiber, is heated to a temperature intermediate to the boiling point of the solvent and room temperature, is mixed with at least five volumes of solvent free gas heated to the approximate boiling point of the solvent at a point close to the fiber source, is passed countercurrent to the freshly spun fiber at the spinneret, and is directed away from the fiber.

2. A method of preparing fibers of acrylonitrile polymers, which comprises extruding a solution of a polymer into a countercurrent stream of air which was introduced at room temperature in contact with the finished fiber, which stream is passed over heating elements in countercurrent contact with the fiber at a temperature below 100° C., which stream is then mixed with at least five volumes of a solvent free gas at a temperature above 140° C., said mixed gas stream being passed countercurrently with respect to the freshly spun fiber at the fiber source.

3. An apparatus for dry-spinning thermoplastic synthetic fibers comprising, a vertical tower, an enclosure attached to the top of the tower, a hinged closure attached to the bottom of the tower having a fiber-exit opening therein, a spinneret mounted in the enclosure and pointed downward toward the tower, conduit means attached adjacent the bottom of the tower for introducing cold gas into the tower, a second conduit means attached to the enclosure adjacent the upper portion thereof for removing gas therefrom, a baffle mounted in the enclosure intermediate the second conduit means and spinneret, a third conduit means attached to the enclosure adjacent the lower portion thereof for introducing hot gas therein, a funnel-shaped member mounted in the enclosure below the spinneret for leading all of the gas stream in parallel countercurrent relationship to the fibers in intimate contact therewith at their source, said funnel-shaped member having its sides diverging away from the spinneret and attached to the wall of the enclosure, a cylindrical baffle means positioned opposite the

6

third conduit means, said cylindrical baffle means extending upwardly into the space formed by said funnel-shaped member, the axes of the cylindrical baffle means and spinneret being coincident, and heating means attached to the wall of the tower.

4. An apparatus as defined in claim 3 wherein the lower end of the tower is spaced from the hinged closure, and wherein the hinged closure has side-walls spaced from the walls of the tower, whereby a passage is formed around the bottom end of the tower, and wherein the first conduit means communicates with said passage.

5. A method of spinning acrylonitrile polymer fiber, which comprises extruding a solution of the acrylonitrile polymer into a countercurrent atmosphere between 140° and 160° C., said atmosphere being comprised of two components, the first component comprising from two to 20 volumes of a gas passing countercurrently with respect to the fiber at a temperature below 100° C., said gas being introduced into the path of the fiber at a point removed from the fiber source, and the second component comprising from 80 to 98 volumes of solvent-free gas at a temperature above 140° C., said gas being introduced into the path of the fiber at a point between the fiber source and the point of introduction of the first gas.

6. A method for spinning fibers from fiber-forming acrylonitrile polymers, which comprises extruding a solution of a polymer of at least 80% acrylonitrile and up to 20% of another monolefinic polymerizable substance into a gaseous medium flowing countercurrent to the direction of extrusion, directing the entire stream of gaseous medium into contact with and immediately adjacent to the freshly formed fibers at the fiber source, said stream being heated to a temperature between 140° C. and 160° C., and passing the fibers in countercurrent relationship to a smaller volume of the said gaseous medium at a temperature below 100° C., said smaller volume being from two to 20% of the said larger volume.

WALTER H. HAMMOND.
LAURENCE C. HOLT.
RODGER L. SCHAEFER.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
1,959,443	Payne et al. _____	May 22, 1934
2,002,066	Taylor _____	May 21, 1935
2,404,714	Latham _____	July 23, 1946
2,404,722	Houtz _____	July 23, 1946