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PROCESS FOR SPINNING ARTIFICIAL FIBERS

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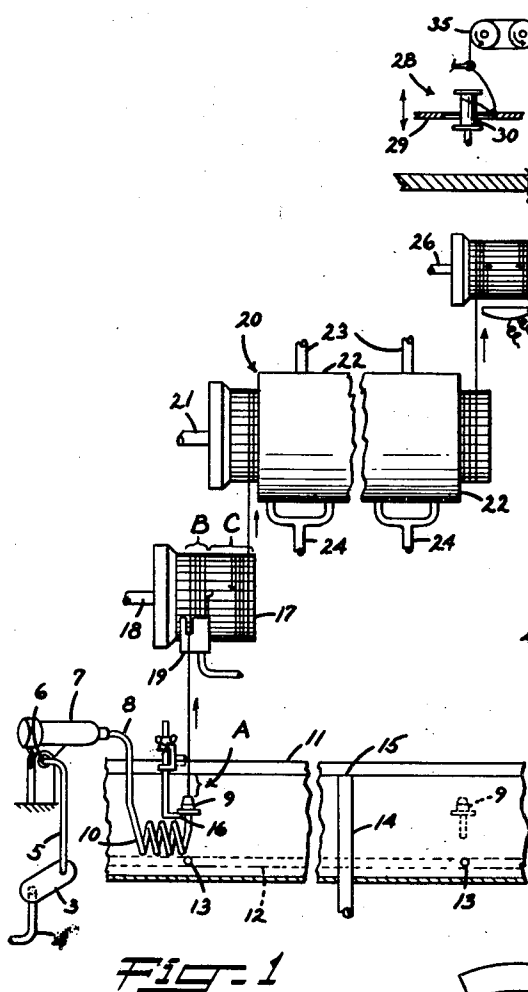


FIG. 1

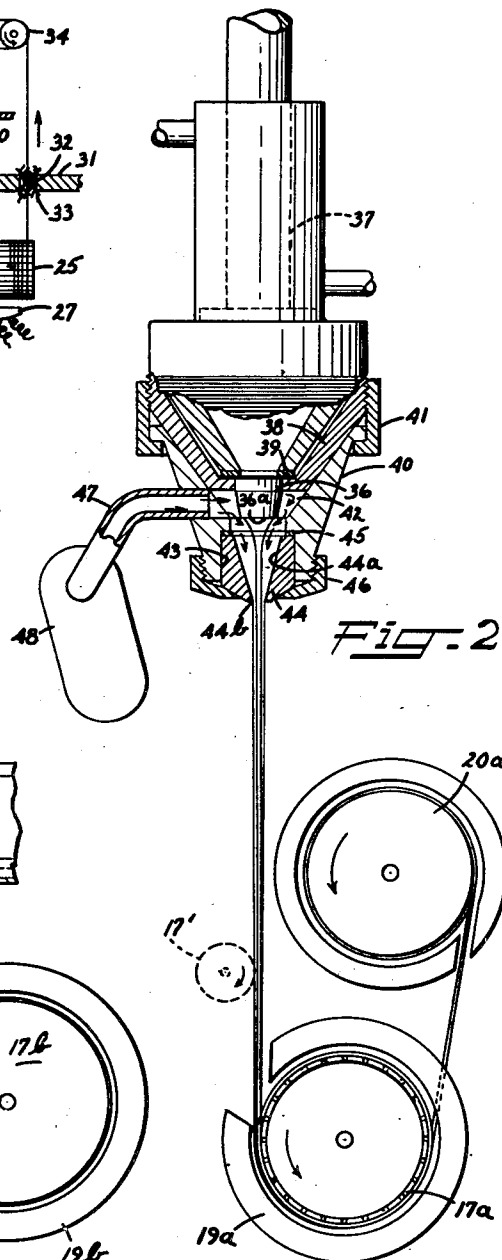
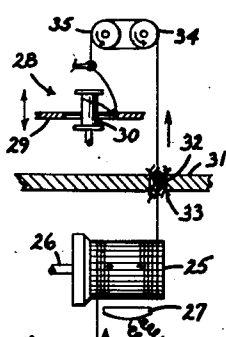


FIG. 2

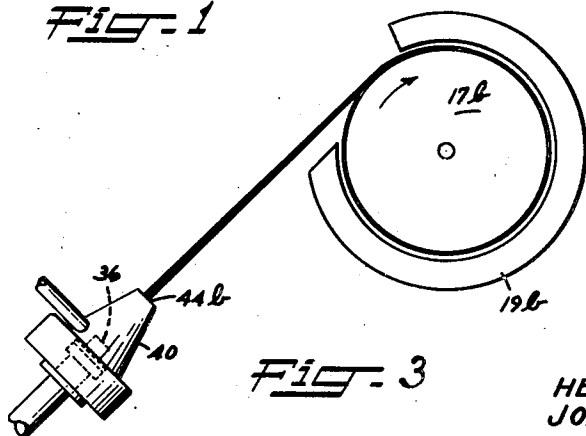


FIG. 3

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PROCESS FOR SPINNING ARTIFICIAL FIBERS

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26 Claims. (Cl. 18-54)

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This invention relates to methods of spinning artificial filaments from viscose by the wet-spinning system in accordance with which filaments extruded through orifices of a spinneret enter an acid coagulating liquid which serves to set up the filaments by removal of solvent and by chemical reaction therewith.

When freshly spun filaments are drawn through a liquid coagulating bath, the resistance imposed thereon by the bath, which may be called bath drag, generally increases exponentially with increase of spinning speed. Increased bath drag reduces the extensibility of the final yarn and also reduces the capacity of the yarn to be stretched during the formative stages of the spinning operation. Generally, the upper limit of stretch without rupture even at spinning speeds of less than 100 meters per minute is not over 50% and frequently much less, such as 35 to 40%. It is possible to reduce this bath drag by causing the coagulating liquid in the bath to flow at the same speed as the yarn but, with normal immersions of the order of 10 to 40 inches or more in length, it is difficult to control the liquid flow so that it is truly uniform along the entire length of the path of the yarn at all times during the spinning operation. Non-uniformities at this stage cause non-uniform yarns subject to dyeing irregularities which are particularly objectionable.

To obtain a higher stretch and resulting higher strength in the yarn without resorting to special control of the flow of coagulating bath, it has been the general practice heretofore to use a high proportion of zinc salt in the bath of about 4% or more. This seems to toughen the yarn, apparently by producing a thick skin thereon, but it is then necessary to soften or plasticize the yarn, such as by the application of a hot water bath of 50° C. to almost 100° C. temperature, before the yarn can be subjected to the higher stretches desired without rupture. Unless the yarn is carefully handled in the plasticizing bath by complete immersion under conditions of accurate control and distribution of the temperature along the yarn path therein, the stretching stage provides an additional source or cause of irregularities in the properties of the final yarn, and particularly the dyeing, strength, and extensibility characteristics thereof. The use of 4% or more zinc salt in the coagulating bath also has the disadvantages of being expensive and rendering the coagulating bath replenishing and recovery systems difficult, chiefly because of the waste disposal hazard involved.

In general, therefore, the conventional spinning procedures employed allow the filaments to be subjected to variable stresses by the coagulating liquid before the filaments are toughened by sufficient setting or coagulation to be insensitive

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to such variable influences so that the final physical properties of the filaments are non-uniform, and this non-uniformity exists along the length of each individual filament as well as between the several filaments issuing from the same or different spinnerets.

All of these non-uniformities increase in geometrical proportion when spinning speed increases arithmetically. When stretch is applied to filaments which have non-uniformities introduced during initial coagulation, the variations are generally accentuated. It is a primary object of the present invention to provide a continuous spinning process which can be executed with a minimum of variation, especially in the initial stages of coagulation or setting through that of stretching when such a step is employed, so that higher speeds of spinning can be used, even for the production of stretched high tenacity filaments, without serious impairment of quality.

In accordance with the present invention, it has now been discovered that it is possible to produce in a practical manner artificial filaments of regenerated cellulose which are markedly improved in respect to strengths, both wet and dry, extensibilities, wet and dry, and dyeing, particularly in respect to the uniformity of such characteristics among the several filaments issuing from a single spinneret as well as along the length of each filament and even among the filaments issuing from several spinnerets, by using a viscose preheated to a temperature of 35° C. to 65° C. and properly controlling the action of an acid coagulating liquid having a temperature between 40° C. and 70° C. upon the freshly extruded filament-forming streams. In accordance with the present process, the filaments can be highly stretched up to 70%, 80% or more to produce high tenacity in the final yarns without introducing irregularities therein, yet the coagulating bath need contain no more than ½ to 1½% of zinc salt and no plasticizing bath is needed to assist the stretching.

In general, the new process involves extruding the viscose into an acid regenerating bath at a low "jet stretch" factor of 0.2 to 1.25 and preferably less than 1 (such factor being herein defined as the ratio of the linear speed of the yarn at the rotor which draws it from the spinneret to the linear velocity of extrusion) and inducing, at a short distance not over five centimeters from the spinneret, a small stream or jet of the coagulating liquid to move midair along with the freshly extruded filament bundle through a straight path at substantially the same speed as the filaments while avoiding irregular disturbances resulting from part of the liquid running back upon or ahead of, or dripping off, the traveling filament-liquid stream during the initial stage of coagulation within 15 centimeters

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of the spinneret, and preferably avoiding such disturbances until such stream reaches a predetermined stage after which removal of the liquid is accomplished in a uniform manner and under conditions subject to control. After liquid removal, any remaining reactive portion of adherent or absorbed coagulant is consumed or brought to a condition of or approaching equilibrium with respect to the filaments. At the end of this stage, the substance of the filaments is about 50% to 70% regenerated, but preferably less than 60% regenerated. Before 70% regeneration is exceeded, and preferably before 60% regeneration is exceeded, a stretch of at least 35% is applied in one or a plurality of stages.

A small but substantial quantity of liquid will adhere as a body to the filaments when they are removed from the coagulating bath so that substantially all of such adherent liquid will travel with the filaments and at the same velocity as the filaments over considerable distances through the air without any noticeable change of velocity regardless of the direction of the flight through the air. The amount of liquid that can thus adhere to the filament bundle and be sustained and moved along bodily with it, in spite of gravitational and air friction forces depends on a number of factors including the particular composition of the filament-forming viscose material, the number of filaments making up the bundle, and the nature of the coagulating liquid, particularly its surface tension, viscosity and tendency to adhere to the material constituting the filaments. The maximum amount that can thus travel with the filaments is not inconsiderable, however, and it has been found that, in general, much less than the maximum amount will in a relatively short passage of 15 to 75 centimeters through midair, even at spinning speeds higher than conventional, set up the filament-forming streams of preheated viscose to a sufficiently firm condition that the filaments leaving this stage of production can stand up under all the customary subsequent treating stages without being unduly sensitive to irregularities therein.

In order to limit the amount of liquid traveling with the thread, the spinneret may be disposed (1) with its orifices discharging upwardly at a predetermined depth of not over 5 centimeters below the constantly maintained level of a bath of coagulating liquid or (2) within a chamber to which the coagulating liquid is supplied, which chamber is provided with an opening in front of the spinneret face through which the filaments accompanied by the liquid are withdrawn in any direction. This opening may be of sufficiently small diameter to limit the amount of liquid passing out of the chamber with the filaments. Depending upon the nature of the material forming the opening and the precise contour at the opening, the diameter of the stream issuing may increase or decrease somewhat from the diameter of the opening and accordingly the selection of the size of the opening should take this into account. The chamber about the spinneret should be kept filled with the coagulating liquid which involves the feeding of such liquid thereto at a rate sufficient to equal the rate of withdrawal by the filaments. In the vicinity of the opening, the contour of the walls about the passage or zone within the chamber is preferably streamlined to assure viscous flow of the liquid as it approaches the opening immediately before passage there-through. This can be adequately met by gradu-

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ally tapering the inside wall or walls of the chamber toward the opening.

In order to prevent irregularities arising from variations in the effective immersion depth of the spinneret, the holes in the spinneret should be spaced apart not over about 0.3 mm. (between centers of the adjacent orifices in a given row whether straight or circular or between the center lines of adjacent rows). This precaution is not necessary when good control of the immersion depth is maintained.

The accompanying drawing is illustrative of the spinning processes of the present invention.

Figure 1 is a front elevation of a machine for executing the invention,

Figure 2 is an end elevation of a modification having an axial section through a spinneret assembly which may be used in performing the invention, and

Figure 3 is an elevation showing another spinning system.

In Figure 1, a gear pump 3 is connected, as by line 4, to a source of supply of viscose which may have the usual "cellar" temperature of about 20° C., and by line 5 to the swivel joint 6 of a candle filter 7 which is connected by a rounder 8 to a spinneret 9. The rounder comprises a coiled section 10 which constitutes a heat-interchange section for transferring heat from the coagulating bath within the receptacle 11. The coiled section 10 is below the spinneret so that when the spinneret is swung into the bath the coil is also submerged. On the other hand, when the spinneret is taken out, the coil also is removed so that there is no heating of the viscose in the rounder when the spinneret is taken out of the bath as is the practice when the pump 3 is stopped for any reason.

Coagulating bath liquid is supplied to the receptacle 11, which may be a trough extending the length of the machine for receiving a plurality of spinnerets at spaced points therealong, by means of a pipe 12 having one or more outlets 13, each of which may be disposed in the vicinity of or below a corresponding spinneret. One or more discharge pipes 14 has or have an overflow opening 15 fixed at a predetermined height to determine the level of the liquid in the bath. The receptacle 11 may, however, be relatively short, such as of a length, width and depth to conveniently contain a single rounder, entrance connection 13, and overflow pipe 15 associated with each individual spinning station.

Adjustable detent means 16 may be provided to assure the proper positioning of the spinneret so that the immersion depth A of the orifices is the value desired, not over 5 centimeters in any event.

Above the receptacle there is a thread-advancing thread-storage device, such as a reel 17 which is rotated on the shaft 18. The filament bundle passes upwardly from the spinneret through the bath a distance A not over 5 centimeters and preferably only 1 to 3 centimeters, and then mid-air a distance of 15 to 75 centimeters, and preferably 20 to 35 centimeters, to the receiving end of the reel 17, carrying a certain amount of coagulating liquid along. This liquid is thrown downwardly off the thread by centrifugal force and gravity combined into a receptacle 19 below the reel. This discharge is effected in from two to six turns about the reel in the section thereof designated B.

After the loosely adhered liquid is thrown off, any remaining reactive portion of the residue is

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partially consumed in the storage section C of the reel 17, the number of turns therein being dependent on the filament denier and on the specific constitution and physical characteristics of the coagulating liquid. The purpose of this section of the reel is to consume the remaining acid as much as possible which serves to advance the regenerating process to a condition in which the cellulose xanthate of the filaments has been regenerated between 50 and 70%, but not over 70%, in terms of combined or xanthate sulfur content.

From the section C of reel 17, the filament bundle proceeds to a reel 20 which is driven on a shaft 21 at a peripheral speed which is the same or greater than that of reel 17 depending on whether or not and what amount of stretch it is desired to apply to the filaments. No plasticizing bath need be applied to the filaments to facilitate the stretching at this stage unless it exceeds 80%.

On reel 20 or on subsequent reels, the filaments are processed by appropriate treating liquids, such as dilute acid at 60 to 85° C. for completion of regeneration, aqueous sodium sulfide for desulfurizing, water rinses, hypochlorite bleach, antichlors, soft finishes, etc. This processing may be advantageously effected in the manner and by the means described in the copending application of Richard W. Stanley and William B. Lowe, Serial No. 504,664, filed October 2, 1943, though any other manner of continuous processing may be used instead. The drawing shows the initial and final liquid processing sections comprising the deflecting jacket 22, which closely surrounds the reel and splashes the liquid thrown off the reel back to it, a liquid feed pipe 23, and liquid discharge means 24.

From the liquid processing section, the yarn proceeds upwardly to a drying section comprising a thread-advancing device 25 driven on shaft 26 and associated with heating means 27 which, for example, may be a steam or electric heating coil.

From the drying section, the yarn passes to a suitable collecting device which may wind and/or twist the filament bundle during collection. A ring twister 28 is shown comprising the ring rail 29 and the bobbin 30. The collecting devices, and, if desired, the drying devices may be on a separate floor 31 above that of the spinning and liquid processing sections so that the final product runs no risk of contamination with any mists or drippings of liquids which may be present in the spinning and processing sections. The yarn passes through a suitable opening 32 in the floor which may be provided with a seal 33 of fur, felt, or the like. Guide rolls 34 and 35 are disposed directly above the opening 32 and the collecting device 28 respectively to properly direct the yarn from the drying to the collective device.

In the embodiment of Figure 2, each spinneret is advantageously disposed within an individual chamber to which the coagulating liquid is supplied, though it is to be understood several spinnerets may be disposed in a common chamber. The spinneret 36 is held in place upon a viscose supply pipe 37 by means of the coupling 38, a gasket 39 being present to provide a seal. A housing member 40 is held in place upon the coupling member 38 by means of the sleeve 41 which threadedly engages the outer periphery of coupling 38. The member 40 contains the chamber 42 and has a recess 43 within which is fitted a guiding element 44 having a tapering bore 44a terminating in an orifice 44b. A gasket 45 is

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seated between the member 44 and the shoulder at the back of the recess 43 and the orificed member 44 is held in place by a coupling sleeve 46 which threadedly engages the outer periphery of the housing member 40.

The coagulating liquid is supplied to the chamber 42 by means of the conduit 47 which may be the discharge conduit of a metering pump 48 such as a gear pump. The conduit 47 fits into the housing member 40 and is arranged to direct the coagulating liquid around the body of the spinneret 36.

As shown, it is preferable, when spinning viscose into acid regenerating baths which develop gases during the spinning to have a portion or even all of the coagulating liquid stream discharged by the conduit 47 directed across the face 36a of the spinneret in order to prevent accumulation of bubbles on the spinneret face. The arrows generally indicate the flow of the coagulating liquid around the spinneret and then into the region immediately in front of the spinneret face. The inner end wall of the member 44 projects inwardly of the adjacent inside wall of the housing and this projecting ledge or shoulder assures that the liquid flows from all sides of the outer periphery of the spinneret toward the center of its face. As the liquid flows inwardly along the face, it contacts with the filaments leaving the several orifices within the spinneret, sweeps gas bubbles from the face, and joins with the filaments in the main current of flow axially of the spinneret and of the tapered bore 44a in the guiding member 44. The transverse flow of the coagulating liquid over the face of the spinneret, though shown as proceeding from all points of the spinneret face periphery, may proceed from one side only of such face so that the transverse flow may deflect the filament-forming streams toward the other side of the spinneret face, as is shown in the copending patent application of Roberts Serial No. 698,437, filed September 21, 1946.

The size of the opening 44b may be preselected, as more particularly described hereinafter, to limit the diameter of the stream or jet of liquid traveling with the filaments from the spinneret assembly. The guide element or nozzle 44 is preferably made of material which is not only resistant to corrosion, erosion, and abrasion, but exerts but little abrading action on any filaments that may occasionally rub thereagainst during the initial lacing or threading operations. Glass, porcelain, agate, lava, and artificial materials of the latter type, such as synthetic sapphire, may be used to advantage for this fitting. The tapered passage 44a merging into the opening 44b may have any length such as to make the distance from 44b to the spinneret 5 cms. or less, but is preferably relatively short so that this distance is 1 to 3 cm. or less.

The spinneret assembly may be arranged to discharge the liquid carrying the filament bundle in any direction from vertically downward to vertically upward. Regardless of the direction taken by the filaments during spinning, i. e., downwardly or upwardly or at an inclination to either direction, the control of the coagulating liquid supplied to the chamber may be accomplished either by means of a metering pump such as a gear pump or by the maintenance of a predetermined pressure upon the liquid supplied to the chamber. When dependence is placed upon a predetermined pressure rather than upon a metering pump, it then

becomes more important to predetermine the diameter of the opening 44b in order to limit the amount of liquid issuing with the filament bundle. Regardless of which system is employed for controlling the supply of coagulating liquid to the chamber, it is of paramount importance to keep the chamber full of coagulating liquid at all times during spinning and also to provide a gradual taper along the bore 44a approaching the opening 44b. This taper assures substantially viscous flow of the coagulating medium through the guiding member 44 and into the free flight or course through the air. Some turbulence may be present in the vicinity around the face of the spinneret where the coagulating liquid sweeps transversely or the filaments and then must change direction to flow through the member 44, but is preferably minimized by forming the parts, such as the member 44 and the spinneret body, with smoothly rounded contours. Whatever small amount of turbulence there may be at any part of the passage followed by the coagulating liquid through the chamber, the fact that the liquid is continuously supplied at a constant rate assures substantially uniform treatment of the filaments within such chamber.

The present invention, however, is vitally concerned with what occurs after the filament-containing coagulating liquid stream or jet leaves the spinneret assembly up to the time of the removal of the excess liquid from the filament bundle. If the liquid carried by the filament bundle begins to flow back down the bundle in the case of upward spinning, irregularities immediately occur which influence the properties of the filaments. Thus, as the liquid starts to flow back down the liquid-carrying filament stream, it disturbs the upward traveling body of liquid and in a fashion characteristic of liquid, a more or less irregular wave pattern is generally set up by such disturbance. The result of such a disturbance is inherently the production of an irregular filament. Such irregularity in the filament may not always be detectable in terms of its diameter but it does show up in variations of strength, extensibility and dye receptivity along the filament. Similarly, in downward spinning, the overtaking mass of liquid similarly sets up a disturbance having a wave pattern of irregular character which again influences the filaments irregularly. When the direction of spinning is horizontal, or either upward or downward at an inclination to the vertical, part of the liquid may drip off the course of the filaments at irregularly varying distances from the spinneret assembly.

All of these irregular occurrences are avoided in accordance with the present invention by limiting the amount of liquid in the jet accompanying the filament bundle to the amount which is able to cohere as a unit and to adhere bodily to the given filament bundle throughout the free flight in midair in the particular direction at which spinning occurs without the back flow, overtaking, or dripping so that the liquid at any point along the bundle as it travels from the spinneret assembly has substantially the same linear velocity throughout its cross section as the filaments traveling therein. It is a relatively simple matter to determine when all of the liquid travels at the same speed as the filaments since, when this condition is present, the diameter of the stream remains constant from a point near the exit opening of the spinneret assembly all the way to the surface which removes the liquid from the filaments. The amount of liquid that can be

carried in this fashion depends upon the particular constitution of the viscose spinning material and the coagulating liquid, but in general, it has been determined that a safe upper limit of load is 0.001 gram per centimeter per filament for bundles containing up to about 300 filaments and optimum results are obtained if the load per filament is not over 0.0006 gram per centimeter. The latter limit is applicable to bundles having any number of filaments such as 1200. In the spinning of viscose in a dilute acid aqueous coagulating bath having a specific gravity of 1.3 to 1.4, it has been found that the limit of load is reached when the filament-containing liquid stream has a diameter of 3 mm. and optimum results are obtained if the diameter does not exceed 2 mm.

The amount of liquid in the jet containing the filaments may be controlled in various ways. The size of the opening 44b can be predetermined to limit the diameter of the liquid jet issuing and the velocity of the jet can be controlled by preselecting the pressure exerted upon the liquid in the chamber 42, or by the operation of the metering pump, depending upon which device is employed. When operated in this fashion, the size of opening may vary in a practical fashion between about 0.5 mm. to 3.0 mm. in diameter though lesser diameters not exceeding 2 mm. are preferred.

When spinning upwardly, some reliance may be placed upon the natural tendency of the filament bundle to carry the liquid with it out of the opening, in which event the diameter of the opening can be much larger than and even several times the above-mentioned 3 mm. limit, but this is satisfactory only when the distance from the face of the spinneret to the liquid level in the exit opening in the chamber is not over 3 cm. and is preferably 2 cm. or less. The filament-containing jet of liquid issuing in this case acquires its velocity chiefly from the filaments being withdrawn from the assembly. The filament bundle itself, in this case, can be used to control the diameter of the liquid stream, and this can be effectively done by varying the spacing between the orifices of the spinneret. When there is used a spacing approaching 0.3 mm. between centers of adjacent orifices in a row and between centers of adjacent rows, a relatively larger diameter of liquid stream will move away with the filaments, whereas a closer spacing, such as 0.15 to 0.2 mm. between centers of adjacent orifices in a row and between centers of adjacent rows, provides a smaller diameter, and as indicated above, the smaller diameter streams are preferred. The closer spacing becomes more advantageous as the number of filaments in the bundle becomes larger.

As in the embodiment of Figure 1, the midair travel of the filaments from the orifice 44b in both Figures 2 and 3 to the reels 17a or 17b respectively should be between 15 and 75 centimeters, and is preferably 20 to 30 centimeters.

In accordance with the present invention, it is desirable to remove the liquid in a controlled manner and this is preferably done by contacting the filament-containing stream with a surface having a linear speed substantially the same as that of the filaments, such as that of the reel 17 of Figure 1 or that of the corresponding rotating member 17a of Figure 2. Such surface may be inclined to one side of the running filaments so that the liquid runs down the surface away from the filaments as they bear against the surface. Or, the surface may be a porous

one, such as that of a perforated or woven screen cylinder or endless belt so that the liquid flows to the interior thereof. Alternatively a rotary screen or drum 17' (such as shown in Fig. 2) having a linear peripheral speed the same as that of the filaments may be lightly pressed against the straight course of the filaments before they reach the thread-advancing device 17 or the surface 17a. The liquid may be sucked through the screen or removed by the drum and wiped off the drum at a point away from that in contact with the filaments. Again, the surface may have a radius such that it may rotate at a sufficiently high speed (though still the same linear speed as that of the filaments) to throw the liquid off the filament bundle by centrifugal force as the filament bundle travels around such surface. This action is specifically shown in both Figures 1 and 2.

Figure 2 is illustrative of the present invention in which spinning is performed in a vertical and downward direction. As shown, the filaments extruded through the orifices of spinneret 36 within the chamber in housing 40 which is supplied with coagulating liquid by the conduit 47 leave the opening 44b in a stream of liquid traveling therewith at substantially the same speed. The filaments pass directly along a straight path from the opening 44b to a rotating generally cylindrical surface 17a, such as that of a screen or a thread-advancing device, such as a reel. The surface 17a is driven at any desired spinning speed from 75 meters upwardly and in accordance with the present invention, it has been found that spinning speeds as high as 200 to 350 meters per minute can be employed with satisfactory production of filaments. When the filaments first reach the peripheral surface 17a, they are pressed thereagainst and the liquid is either thrown off by centrifugal force into the gutter 19a therebelow or if a screen or other permeable surface is used at 17a, the liquid is preferably sucked through, as by a vacuum or reduced pressure. Since all of the liquid carried with the filaments travels at the same speed throughout the passage from the spinneret to the surface 17a, and on 17a, the liquid is removed uniformly from each point of the filament bundle as such points reach the surface in succession, and there is no opportunity for the flow of liquid back down the filaments and the irregular disturbances developed thereby.

From the surface 17a, the bundle of filaments from which the liquid has been removed and which has been brought to a substantially stagnant condition may pass upwardly to a thread-advancing device 20a and then through a system the same as shown in Figure 1 to produce the final dried filaments. The filaments are ultimately collected either in the form of an untwisted or twisted package. For example, they may be collected in a centrifugal pot in the fashion conventional in the viscose spinning process; or they may be collected by winding on a bobbin without twisting; or they may be collected on twistors of the cap, flyer, or ring type.

Figure 3 shows a modification in which the filaments leaving the opening 44b in the housing 40 around the spinneret 36 pass upwardly at an inclination to the vertical and in this embodiment, the filaments in the jet of the coagulating liquid proceed directly to a thread-advancing reel 17b rotating as in Figures 1 and 2 at a sufficiently high speed to throw the liquid from the helix of thread carried thereon. As shown a guard member 19b is provided around the bottom zone

of the reel. This guard member receives the liquid thrown off by centrifugal force and discharges it through a drain 19b connected to the bottom thereof. Beyond this throw-off zone of the reel, the filaments proceed through a storage zone in which they become 50 to 70% regenerated.

The present invention requires a number of critical factors working together to produce the improved filaments and fibers at high spinning speeds of 100 meters per minute and especially between 150 and 350 meters per minute. At such high speeds, it is vitally important that interruptions of spinning be reduced to a minimum. For this reason it is most advantageous to spin at a jet stretch of less than 1 and specifically from 0.2 to 0.9 since it has been found that incrustation of the orifices is greatly reduced under such conditions, apparently by virtue of the velocity of the viscose itself passing through the orifices. This can be further improved by incorporating into the viscose and coagulating bath, such assistants as the Carbowaxes (e. g. an ethylene oxide polymer of 4000 molecular weight in the bath and/or such a polymer of 6000-9000 molecular weight in the viscose), cation-active compounds, such as lauryl pyridinium chloride, the compounds of the Collins Patents 2,359,749-50, and so on.

It also requires, when a high tenacity product is desired, that a viscose of a temperature between 35° C. and 65° C. (preferably below the temperature of the spinning bath) be extruded into an acid regenerating bath containing between 1/2 to 1 1/2% zinc sulfate, and that a stretch of at least 50% be performed on the filaments in a condition of not over 70% regeneration.

Regardless of whether normal or high strength filaments are produced, the extrusion of the heated viscose through orifices which are close together (not over 0.3 mm. apart center to center in a row or between adjacent rows) into a short body of coagulating liquid (not over 5 cms. from the spinneret face) from which the filament bundle takes a narrow stream traveling midair a distance of 15 to 75 centimeters at the same speed as they do serves to prevent non-uniformities in filament properties from being derived from this stage of operations. This is followed by uniformly removing the stream carried by the filament bundle at a definite point in its travel directly from the spinning bath.

The present invention, however, not only controls the flow of coagulating liquid so that the amount carried by the filaments is not so excessive as to develop back flow and dripping and other irregular disturbances but it also economizes on the amount of coagulating liquid needed for complete set-up. Furthermore, it assures that the coagulating liquid which makes contact with the filaments immediately after the material leaves the spinneret orifices has a definite composition which is constant throughout the period of spinning. Thus, each portion of the length of each filament is in contact with a coagulating bath having a definite constitution at any point of its passage between the spinneret orifice and the stage at which coagulating liquid is removed. Fresh coagulating liquid of the predetermined constitution makes the first contact with the filaments issuing from the spinneret and such liquid, contaminated as a result of contact with the filaments, is not allowed to tarry at the spinneret face to react with subsequently issuing filaments but instead is continuously withdrawn with the filaments so that fresh liquid replaces that at

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the spinneret face in a continuous fashion. This action is assured by the provision of the shallow immersion of Figure 1 or of the relatively small chamber about the spinneret (in Figures 2 and 3) which is kept filled with coagulating liquid and is rapidly withdrawn with the filaments. Since the coagulating liquid always has a velocity in the direction of flow of the filaments from the spinneret face, there is very little bath drag to exert tension upon the filaments as they pass from the spinneret to the stage at which the coagulating liquid is removed. In addition, the direct travel of the filaments from the spinneret face to the stage at which the liquid is removed in a controlled and uniform fashion assures that the characteristics of the filaments are as uniform as possible. Furthermore, when stretching is applied for the production of high tenacity filaments, it is applied at a stage in which the filaments, though still not completely regenerated, are changing at a slow rate because the residue of coagulating bath in the immediate vicinity of the filaments has been consumed. The present invention is particularly advantageous in high speed continuous spinning and for this purpose, its form shown in Figures 2 and 3 makes use of controlled pressure or a metering pump for delivering the coagulating liquid to the chamber about the spinneret at a predetermined rate, in combination with the preselection of the diameter of the opening in the chamber through which the filaments are discharged for controlling the diameter of the stream issued therefrom so that in effect, the liquid discharged from that opening serves to carry the filaments rather than the converse. When operating in this fashion, a high speed jet of liquid issues and assists the withdrawing device in carrying the filaments thereto.

The following examples are illustrative of the invention and the percentages therein are given by weight. The total quality index or factor referred to is the sum of the products obtained by multiplying the dry tensile strength by the dry extensibility and the wet tensile strength by wet extensibility. This quality index will refer to the sum of the products of these values unless otherwise indicated.

Example I

A viscose having 7% cellulose and 6% caustic soda and a salt point (NaCl) of 5.0 was spun at a temperature of 35° C. into a coagulating bath containing 10% H₂SO₄, 1% ZnSO₄, and 16% Na₂SO₄ at a temperature of 60° C. The spinneret had 60 holes of 0.0025 inch diameter bore and spaced apart 0.006 inches (0.152 mm.) and its face was ½ inch from the start of midair travel of the filament stream, which extended over a distance of 33 cm. The spinning was under conditions of 0.86 jet stretch and a draw-off speed of 165 meters per minute at the first reel (17, 17a, or 17b of Figs. 1, 2, and 3 respectively). The yarn was stretched 37% between this reel and the next, continuously processed, and dried, and collected at 215 meters per minute. The yarn was permitted to shrink a total amount between 4 and 5% as it proceeded to the drier and on the drier. The yarn was 150 denier and had a residual shrinkage of 3.01%. It had a dry strength of 2.25 g./d., a dry extensibility of 20%, a wet strength of 1.18 g./d. and a wet extensibility of 34%. The total quality factor was 85.

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Example II

A viscose having the same characteristics as that of Example I was spun under the same conditions as in Example I except that its temperature was 50° C. instead of 35° C. The product had a dry tensile strength of 2.24 grams per denier, a dry extensibility of 20.5%, a wet tensile strength of 1.08 grams per denier and a wet extensibility of 35%. Its total quality factor was 84.

Example III

A viscose having the same characteristics as that of Example I was spun under the same conditions as in Example I except that it had a temperature of 55° C. instead of 35° C. The product had a dry tenacity of 2.34 grams per denier, a dry extensibility of 18.8%, a wet tenacity of 1.25 grams per denier and a wet extensibility of 33.7%. It had a quality index of 86 and it had a residual shrinkage of 3.96%.

Example IV

A viscose having the same characteristics as that of Example I was spun under the same conditions recited therein except that its temperature was 45° C. instead of 35° C. The product had a dry tenacity of 2.37 grams per denier, a dry extensibility of 18%, a wet tenacity of 1.25 grams per denier, and a wet extensibility of 32.5%. Its quality index was 83 and it had a residual shrinkage of 3.77%.

Example V

Viscose having the same characteristics as that of Example I except that its temperature was 55° C. was spun under the same conditions as in Example I except as follows: the spinneret face was ¾ inch from the start of midair travel; bath temperature was 65° C.; the draw-off speed of the first reel was 157 meters per minute; there was 45% stretch between the first and second reels. The product was a 148 denier yarn having a dry tenacity of 2.54 grams per denier, a dry extensibility of 17.6%, a wet tenacity of 1.39 grams per denier, a wet extensibility of 28.7%, a total quality factor of 84, and a residual shrinkage of 3.03%.

Example VI

Viscose made of 100% wood pulp having the same characteristics as that of Example I except that its salt point was 4.75 and its temperature was 65–67° C. was spun under the same conditions as in Example I except as follows: the spinneret face was ¼ inch from the start of midair travel; the coagulating bath contained 10% sulfuric acid, 1.5% zinc sulfate, and 17% sodium sulfate and had a temperature of 75° C.; the draw-off speed of the first reel was 156 meters per minute; there was 50% stretch between the first and second reels. The product had a dry tenacity of 2.43 grams per denier, a dry extensibility of 19%, a wet tenacity of 1.44 grams per denier, a wet extensibility of 34.5%, a total quality factor of 95.8, and 3.75% residual shrinkage.

Example VII

A viscose having the same characteristics as that of Example VI was spun under the same conditions as in Example VI except that the coagulating bath contained 10.5% sulfuric acid, 4% zinc sulfate, and 18% sodium sulfate. The product had a dry tenacity of 2.4 grams per denier, a dry extensibility of 19%, a wet tenacity of 1.5 grams per denier, a wet extensibility of 34.5%, and a total quality factor of 96.6.

Example VIII

A viscose of high alpha cellulose content made by using a mixture of 70% cotton pulp and 30% wood pulp and having the same characteristics as those specified in Example VII except that its salt point was 5.95 was spun under the same conditions as specified in Example VII except for the following: the draw-off speed at the first reel was 134 meters per minute, and the stretch between the first and second reels was 70%. The product had a dry tenacity of 2.94 grams per denier, a dry extensibility of 15.5%, a wet tenacity of 1.83 grams per denier, a wet extensibility of 23.5%, a total quality factor of 88.5, and a residual shrinkage of 3.81%.

Example IX

A viscose having the same characteristics as specified in Example VIII was spun under the same conditions as in Example VIII except for the following: the draw-off speed at the first reel was 123 meters per minute, the yarn was stretched 90% between the first and second reels while being subjected to an aqueous plasticizing bath containing 2.5% H_2SO_4 , 0.2% $ZnSO_4$, and 3% Na_2SO_4 at 85° C. which corresponds in composition and temperature to the first bath to which the yarn is subjected on the second reel in each of the preceding Examples I to VIII as well as in this one. This treatment on the second reel completes the regeneration which is followed by a wash with water, desulfurizing with sodium sulfide, water wash, a bleach with sodium hypochloride or the like, then with hydrochloric acid, with a final wash, and then with a soft finish, such as sodium oleate, sorbitan monopalmitate modified with 3 to 20 mols of ethylene oxide, polyvinyl alcohol, or mixtures of such compounds. The product had a dry tenacity of 3.44 grams per denier, a dry extensibility of 13.5%, a wet tenacity of 2.18 grams per denier, a wet extensibility of 18.5%, and a total quality factor of 86.7.

It is to be understood that changes and variations may be made without departing from the spirit and scope of the invention as defined in the appended claims.

We claim:

1. The method of producing artificial filaments comprising continuously extruding viscose having a temperature of 35° C. to 65° C. through the orifices of a spinneret into an acid regenerating and coagulating liquid having a temperature between 40° C. and 70° C., said extrusion being performed under conditions of jet stretch between 0.2 and 1.25, directing the filament-forming streams in a straight path as they proceed from the spinneret and are coagulated to filaments in the coagulating liquid, imparting a predetermined constant velocity to the filaments as they proceed in said straight path, forming the coagulating liquid at a point not over 5 cm. from the spinneret face into a stream having substantially the same velocity as the filaments and flowing in free flight from this point through the air along the straight path of the filaments a distance of at least 15 centimeters, the amount of liquid in the liquid stream being limited to assure that all of the liquid adheres to and travels substantially at the same velocity as the filaments throughout at least the first portion of said straight path of at least 15 centimeters length, and then continuously removing liquid from the filaments in a uniformly controlled manner at a predetermined point of the straight path beyond said first portion.

2. The method of claim 1 in which the direction of extrusion is upward.

3. The method of claim 1 in which the direction of extrusion is downward.

4. The method of claim 1 in which the direction of extrusion is vertically upward, and the liquid removal is effected by a rotating member from whose surface the liquid is thrown off by centrifugal force.

5. The method of claim 1 in which the direction of extrusion is vertically downward, and the liquid removal is effected by suction through a permeable surface traveling at the same speed as the filaments.

6. The method of producing artificial filaments comprising continuously extruding viscose having a temperature of 35° C. to 65° C. through the orifices of a spinneret into a stream of an acid regenerating and coagulating liquid having a temperature between 40° C. and 70° C. and flowing at gradually increasing velocity in the general direction of extrusion, said extrusion being performed under conditions of jet stretch between 0.2 and 1.25, directing the filament-forming streams of material in a straight path as they proceed from the spinneret and are coagulated to filaments in the coagulating liquid stream; imparting a predetermined constant velocity to the filaments along said straight path; forming the coagulating liquid stream at a point not over 5 cms. from the spinneret face into a jet or stream having substantially the same velocity as the filaments, flowing in free flight from this point through the air along the straight path of the filaments a distance of 15 to 75 centimeters, and having a weight per centimeter of length not over 0.001 gram per filament so as to assure that all of the liquid adheres to and travels substantially at the same velocity as the filaments throughout said straight path, and then continuously removing liquid from the filaments in a uniformly controlled manner at the end of the straight path.

7. The method of claim 6 in which the jet has a diameter not over 3 mm.

8. The method of claim 6 in which the jet has a diameter not over 2 mm.

9. The method of claim 6 in which the jet has a weight per centimeter of length not over 0.0006 gram per filament and a diameter not over 3 mm.

10. The method of claim 6 in which the jet has a weight per centimeter of length not over 0.0006 gram per filament and a diameter not over 2 mm.

11. The method of producing artificial filaments comprising continuously extruding viscose having a temperature of 35° C. to 65° C. through the orifices of a spinneret spaced apart by a distance not over 0.3 mm. into a body of an acid regenerating and coagulating liquid having a temperature between 40° C. and 70° C., said extrusion being performed under conditions of jet stretch between 0.2 and 1.25, directing the filament-forming streams in a straight path as they proceed from the spinneret and are coagulated to filaments in the coagulating liquid, imparting a predetermined constant velocity to the filaments as they proceed in said straight path, forming the coagulating liquid at a point not over 5 cms. from the spinneret face into a stream having substantially the same velocity as the filaments and flowing in free flight from this point through the air along the straight path of the filaments a distance of 15 to 75 centimeters, the amount of liquid in the liquid

stream being limited to assure that all of the liquid adheres to and travels substantially at the same velocity as the filaments throughout said straight path, and then continuously removing liquid from the filaments in a uniformly controlled manner at the end of the straight path by passing the filaments through a helical path at a linear speed sufficient to throw the liquid off the filaments, continuing the passage of the filaments through the helical path beyond the point of liquid throw-off a sufficient distance to allow further reaction of the liquid residue left on the filaments, and then stretching the filaments before they have exceeded a stage of 70% regeneration.

12. The method of producing artificial filaments comprising continuously extruding viscose having a temperature of 35° C. to 65° C., under conditions of jet stretch between 0.2 and 1.25, through the orifices of a spinneret spaced apart by a distance not over 0.3 mm. into a stream of an acid regenerating and coagulating liquid, moving the liquid in said stream away from the spinneret at gradually increasing velocity in the general direction of extrusion, withdrawing the filament-forming streams of material from the spinneret in a straight path at a predetermined constant velocity, forming the coagulating liquid stream at a point not over 5 cm. from the spinneret face into a jet having substantially the same velocity as the filaments and flowing in free flight from this point through the air along the straight path of the filaments a distance of 15 to 75 centimeters, the amount of liquid in the jet being limited to assure that all of the liquid adheres to and travels substantially at the same velocity as the filaments throughout said straight path, then continuously removing liquid from the filaments in a uniformly controlled manner at the end of the straight path by passing the filaments through a helical path at a linear speed sufficient to throw the liquid off the filaments, continuing the passage of the filaments through the helical path beyond the point of liquid throw-off a sufficient distance to allow further reaction of the liquid residue left on the filaments, and then stretching the filaments while they are in a stage of 50 to 70% regeneration.

13. The method of claim 11 in which the jet has a weight per centimeter of length not over 0.001 gram per filament and a diameter not over 3 mm. and the filaments are stretched at least 35% in length without application of a plasticizing medium.

14. The method of claim 11 in which the jet has a weight per centimeter of length not over 0.0006 gram per filament and a diameter not over 2 mm. and the filaments are stretched at least 35% in length without application of a plasticizing medium.

15. The method of producing artificial filaments comprising continuously extruding viscose having a temperature of 35° C. to 65° C., under conditions of jet stretch between 0.2 and 1.25, through the orifices of a spinneret spaced apart by a distance not over 0.3 mm. into a confined passage having a restricted exit opening not over 5 centimeters from the spinneret, continuously directing an acid regenerating and coagulating liquid of predetermined constitution having a temperature between 40° C. and 70° C. through the passage and the opening at sufficient velocity to form a jet of the liquid issuing from the opening, continuously withdrawing the fila-

ments through the opening and directing them with the jet in a straight free flight for a distance of 15 to 75 centimeters at substantially the same speed as the jet, the amount of liquid in the jet being limited to assure that all of the liquid adheres to and travels substantially at the same velocity as the filaments throughout the free flight, then continuously removing liquid from the filaments at the end of the free flight by passing the filaments through a helical path at a linear speed sufficient to throw the liquid off the filaments, continuing the passage of the filaments through the helical path beyond the point of liquid throw-off a sufficient distance to allow further reaction of the liquid residue left on the filaments, and then stretching the filaments before they have reached a stage of 60% regeneration, passing the filaments through a helical path while treating them with liquids to purify and condition them, drying the filaments while continuously passing them through a helical path, and then collecting them by winding.

16. The method of claim 15 in which the jet has a weight per centimeter of length not over 0.001 gram per filament and a diameter not over 3 mm. and the filaments are stretched at least 35% in length without the application of a plasticizing medium.

17. The method of claim 15 in which the jet has a weight per centimeter of length not over 0.0006 gram per filament and a diameter not over 2 mm. and the filaments are stretched at least 35% in length without the application of a plasticizing medium.

18. The method of producing artificial filaments comprising continuously extruding viscose having a temperature of 35° C. to 65° C., under conditions of jet stretch between 0.2 and 0.9, through the orifices of a spinneret spaced apart by a distance not over 0.3 mm. into a confined tapered passage having a restricted exit opening not over 5 cms. from the spinneret and a transverse cross-section along a portion thereof decreasing gradually to, and merging with, the opening, continuously directing an acid regenerating and coagulating liquid of predetermined constitution having a temperature between 40° C. and 70° C. and containing ½ to 5% zinc sulfate through the passage and the opening at sufficient velocity to form a jet of the liquid issuing from the opening, continuously withdrawing the filaments through the opening and directing them with the jet in a straight free flight for a distance of 20 to 35 centimeters at substantially the same speed as the jet, said speed being between 100 and 350 meters per minute, the amount of liquid in the jet being limited to assure that all of the liquid adheres to and travels substantially at the same velocity as the filaments throughout the free flight, then continuously removing liquid from the filaments at the end of the free flight by passing the filaments through a helical path at a linear speed sufficient to throw the liquid off the filaments continuing the passage of the filaments through the helical path beyond the point of liquid throw-off a sufficient distance to allow further reaction of the liquid residue left on the filaments and then stretching the filaments without application of a plasticizing medium before they have reached a stage of 60% regeneration, passing the filaments through a helical path while treating them with liquids to purify and condition them, drying the filaments while continuously passing them through a helical path, and then collecting them by winding.

19. The method of producing artificial filaments comprising continuously extruding a viscose spinning solution having a temperature of 35° C. to 65° C., under conditions of jet stretch between 0.2 and 0.9, through the orifices of a spinneret spaced apart by a distance not over 0.3 mm. into a confined tapered passage having a restricted exit opening not over 5 cms. from the spinneret and a transverse cross-section along a portion thereof, decreasing gradually to, and merging with, the opening, continuously directing an aqueous, acid coagulating liquid having a temperature between 40° C. and 70° C. and containing ½ to 1½% zinc sulfate transversely past the viscose streams at the position of extrusion and then through the passage and the opening at sufficient velocity to form a jet of the liquid issuing from the opening, continuously withdrawing the filaments through the opening and directing them with the jet in a straight free flight for a distance of 33 centimeters at substantially the same speed as the jet, said speed being between 100 and 350 meters per minute, the amount of liquid in the jet being limited to assure that all of the liquid adheres to and travels substantially at the same velocity as the filaments throughout the free flight, then continuously removing liquid from the filaments at the end of the free flight by passing the filaments through a helical path at a linear speed sufficient to throw the liquid off the filaments, continuing the passage of the filaments through the helical path beyond the point of liquid throw-off a sufficient distance to allow further reaction of the liquid residue left on the filaments, and then stretching the filaments without the application of a plasticizing medium when they have reached a stage of 55 to 60% regeneration, passing the filaments through a helical path while treating them with liquids to purify and condition them, drying the filaments while continuously passing them through a helical path, and then collecting them by winding.

20. The method of producing artificial filaments comprising continuously extruding a viscose spinning solution having a temperature of 35° C. to 65° C., under conditions of jet stretch between 0.2 and 0.9, through the orifices of a spinneret spaced apart by a distance not over 0.3 mm., continuously directing an aqueous, acid coagulating liquid having a temperature between 40° C. and 70° C. and containing ½ to 1½% zinc sulfate transversely past the viscose streams at the position of extrusion into a zone surrounding the viscose streams, directing the filaments from the vicinity of the spinneret along a straight path comprising a free flight portion of 15 to 75 centimeters at a predetermined constant velocity, said free flight portion starting at a point not over 5 cms. from the spinneret, passing all of the coagulating liquid contaminated by proximity to and contact with the viscose streams with gradually increasing velocity through said zone away from the spinneret in the same direction as the viscose streams while supporting the filaments thereby until the velocity of the liquid is approximately the same as that of the filaments, the amount of liquid in contact with the filaments along the free flight portion being limited to assure that all of the liquid adheres to and travels substantially at the same velocity as the filaments throughout said portion, said velocity being between 100 and 350 meters per minute, then continuously removing liquid from the filaments at

the end of the free flight portion by passing the filaments through a helical path at a linear speed sufficient to throw the liquid off the filaments, continuing the passage of the filaments through the helical path beyond the point of liquid throw-off a sufficient distance to allow further reaction of the liquid residue left on the filaments, and then stretching the filaments without the application of a plasticizing medium before they have reached a stage of 60% regeneration, passing the filaments through a helical path while treating them with liquids to purify and condition them, drying the filaments while continuously passing them through a helical path, and then collecting them by winding.

21. The method of producing artificial filaments comprising continuously extruding viscose having a temperature of 35° C. to 65° C. through the orifices of a spinneret into an acid regenerating and coagulating liquid having a temperature between 40° C. and 70° C., directing the filament-forming streams in a straight path as they proceed from the spinneret and are coagulated to filaments in the coagulating liquid, imparting a predetermined constant velocity to the filaments as they proceed in said straight path, forming the coagulating liquid at a point not over 5 cm. from the spinneret face into a jet or stream having substantially the same velocity as the filaments and flowing in free flight from this point through the air along the straight path of the filaments a distance of at least 15 centimeters, the amount of liquid in the liquid stream being limited to assure that all of the liquid adheres to and travels substantially at the same velocity as the filaments throughout at least the first portion of said straight path of at least 15 centimeters length, then continuously removing liquid from the filaments in a uniformly controlled manner at a predetermined point of the straight path beyond said first portion, passing the filaments through a path comprising at least one helix, treating them as they pass through a helical portion of the path with liquids, drying them at a subsequent portion of the path, and collecting the filaments.

22. The method of claim 21 in which the direction of extrusion is upward.

23. The method of claim 21 in which the direction of extrusion is downward.

24. The method of claim 21 in which the direction of extrusion is vertically upward, and the liquid removal is effected by a rotating member from whose surface the liquid is thrown off by centrifugal force.

25. The method of claim 21 in which the jet has a diameter not over 3 mm.

26. The method of claim 21 in which the jet has a weight per centimeter of length not over 0.0006 gram per filament and a diameter not over 2 mm.

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