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2,054,852

MANUFACTURE OF ARTIFICIAL THREADS, RIBBONS, AND LIKE MATERIALS

Filed Feb. 18, 1932

FIG. 1

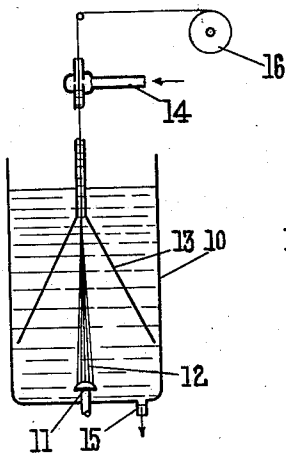


FIG. 2

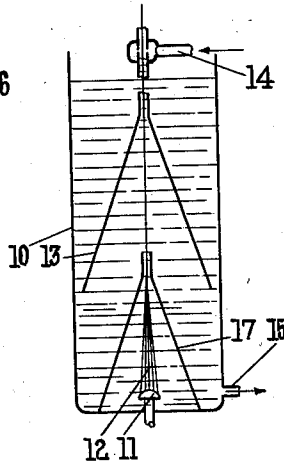


FIG. 3

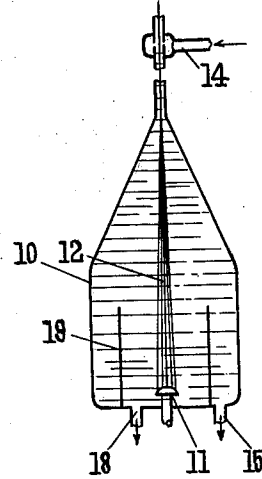
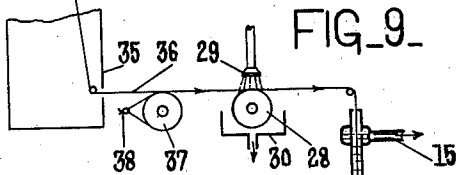


FIG. 9

FIG. 4

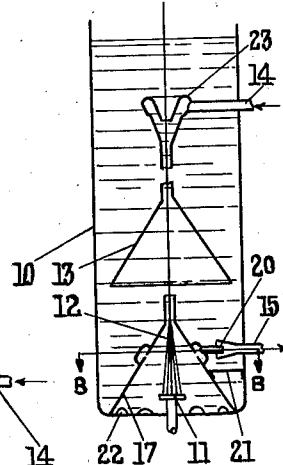


FIG. 8

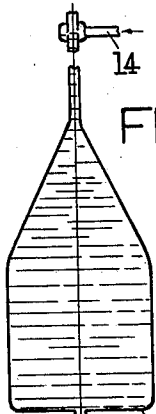


FIG. 6

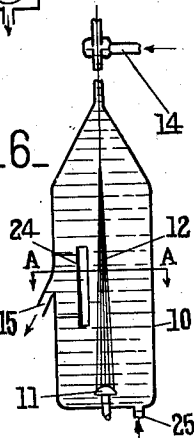


FIG. 7

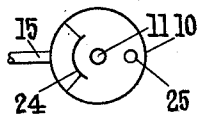
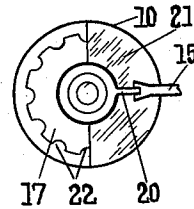


FIG. 5



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MANUFACTURE OF ARTIFICIAL THREADS,
RIBBONS, AND LIKE MATERIALS

Henry Dreyfus, London, England

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In Great Britain March 27, 1931

8 Claims. (Cl. 18—54)

This invention relates to the manufacture of filaments, threads, fibres, yarns, ribbons and the like of cellulose acetate or other derivatives of cellulose, and in particular to a manufacture in which the materials are stretched while passing through a liquid medium.

When filaments, threads and the like of cellulose acetate or other derivatives of cellulose are caused to pass through a liquid medium, by virtue of the relative motion between the materials and the medium, a frictional force is set up which tends to oppose the motion of the materials. In the case of a liquid medium of relatively low viscosity, for example water, the frictional resistance may be comparatively small. A far greater effect however is obtained when liquid media of comparatively high viscosity, for example aqueous solutions of sugars, polysaccharides or other polyhydric alcohols are employed.

I have now found that this frictional effect may be turned to great advantage by causing the liquid to move in such a direction as to retard the motion of the materials to a greater extent than would occur were the liquid at rest. By this means the magnitude and location of the extension which the materials undergo in their course through the liquid medium may readily be controlled. Thus, for example, provided the materials are sufficiently plastic, by imparting to the liquid at a given point along the path of the materials a greater velocity than it possesses elsewhere along the path a relatively great extension may be produced between said point and the point of application of the tension. This ability to control the distribution of extension along the path of the materials is of great advantage, particularly when applied to a coagulating bath employed in the production of the materials by spinning processes. Filaments emerging from a spinning jet increase in strength as coagulation progresses, and it is naturally advantageous to be able to control the process so that relatively great extension occurs at a point where the materials have acquired considerable tensile strength.

According therefore to the present invention filaments, threads, fibres, yarns, ribbons and the like of cellulose acetate or other derivatives of cellulose are caused to pass through liquid media moving in such a direction as to exert a greater drag upon the materials than if the liquid were at rest. The process of the invention may be carried out continuously with the production of the materials by a wet or dry spinning process or

as a separate treatment applied, for example, to fully formed materials. The process of the invention is particularly valuable when applied as a step in the actual production of the materials by a wet spinning process, when for example the coagulating bath itself is set in motion. This aspect of the invention will therefore be described in detail.

The invention contemplates the manufacture of materials made of or containing cellulose acetate or other derivatives of cellulose, for example other esters of cellulose such as cellulose nitrate, formate, propionate or butyrate, ethers of cellulose, for example methyl, ethyl or benzyl cellulose, mixed esters e. g. cellulose nitroacetate, and mixed ethers and ether-esters. The manufacture should be such that the resulting products have a more or less continuous gel structure.

The spinning solution may be made by dissolving the cellulose acetate or other derivative of cellulose in any suitable solvent. As examples of suitable organic solvents for cellulose acetate mention may be made of acetone, acetic acid, and mixtures of ethylene or methylene dichloride with ethyl or methyl alcohol. The spinning solution may contain relatively high boiling solvents or even plasticizers, for instance diacetone alcohol, ethyl lactate or diethyl tartrate. It is of advantage, with a view to obtaining products of more or less continuous gel structure, for the spinning solution to contain solvents or swelling agents for the cellulose derivative, which are less soluble in the coagulating medium than the solvent which constitutes the bulk of the spinning solution.

The percentage of cellulose derivative in the spinning solution may vary within wide limits, but in general concentrations of from 15 to 30%, and particularly about 25%, have been found suitable. Obviously the solution should be neither so dilute that excessive shrinkage occurs, nor so concentrated as to render spinning impracticable. Where extrusion occurs at temperatures above normal, or where the characteristics of the cellulose derivative are such that solutions of comparatively low viscosity are obtained, higher concentrations than are otherwise permissible may be employed.

It is preferable to effect coagulation relatively slowly, for example under such conditions that the materials may leave the coagulating medium in a more or less plastic state. In this way the materials may be obtained in structurally continuous gel form, without the necessity of any further treatment. As described above, coagula-

tion may be delayed by the incorporation of suitable substances in the spinning solution. Alternatively, or in addition, other means may be adopted to decrease the rate of coagulation. For example the coagulating medium may contain solvents or swelling agents for the cellulose derivative. Thus I may employ aqueous coagulants containing thiocyanates, zinc chloride or other inorganic swelling agents for the cellulose derivative, or organic swelling agents, for example diacetone alcohol, acetic acid, formic acid, as described in U. S. Patents Nos. 1,465,994 and 1,467,493, or the relatively strong solutions of solvents or plasticizing agents of U. S. applications S. Nos. 402,785 filed 26th October 1929 and 418,414 filed 3rd January 1930, with or without salts, sugars and the like as described in U. S. application S. No. 469,622 filed 21st July, 1930. For example, a 25% solution of cellulose acetate in acetone which contains also 6% of ethyl lactate, may be spun into a coagulating bath consisting of 49 parts by weight of ethyl lactate, 2 parts by weight of sodium chloride and 49 parts by weight of water.

I prefer to employ aqueous coagulating media such that the materials remain plastic for some distance along their path and may even emerge from the coagulating medium in a more or less plastic condition. Other coagulating media may however be employed. The solution may, for example, be spun into baths containing paraffin oil, petroleum spirit, petroleum ether, toluene or other hydrocarbons, nitrobenzene or other nitroaromatic hydrocarbons, hexachlorethane or other suitable aliphatic or aromatic halogenated hydrocarbons; or animal or vegetable oils, e. g. castor oil, sesame oil or olein may be employed. The coagulating medium must naturally be such that it dissolves at least one constituent present in substantial proportions in the spinning solution, and is a non-solvent for the cellulose derivative. As in the case of aqueous coagulating media, so in non-aqueous media solvents or swelling agents for the cellulose derivative may be present.

It is of very considerable advantage to employ coagulating media of relatively high viscosity. Baths containing a large proportion of mineral acids, e. g. sulphuric acid, mineral acid esters, e. g. ethyl sulphuric acid, or other products resulting from the sulphonation or sulphation of organic compounds, e. g. benzene sulphonic acid, Turkey red oil, Twitchell's reagent and sulphonated brown coal oil are valuable in this respect. The viscosity of the coagulant may be increased by the addition of suitable substances thereto. A large variety of substances is available for this purpose. In the case of aqueous coagulants I have found that valuable results may be obtained by the addition of carbohydrates and other polyhydroxy compounds, e. g. starch, sulphite cellulose waste lye and other poly-saccharides, sucrose, molasses, glucose, lactose, mannitol, dulcitol and glycerol. Thus a spinning solution consisting of 25% of cellulose acetate dissolved in acetone may be spun into an aqueous coagulating bath consisting of 30% by weight of diacetone alcohol, 30% by weight of glucose and 40% by weight of water. The particular substances selected must, of course, be compatible with the other constituents of the coagulating medium.

The spinning solution may be extruded directly into a body of liquid flowing counter to the direction of extrusion. It is however preferable to extrude into a substantially stationary body of liquid and to pass the partly coagulated threads

directly therefrom into the moving liquid in which coagulation is continued or completed. The materials may, for example, be formed by spinning into a bath in which the coagulant is practically at rest, and may be carried thence without passage through the air into a column of liquid moving so as to oppose the travel of the materials. On the other hand, the spinning jets or dies may be situated in a "dead zone" in a body of moving liquid, i. e. in a region in which the coagulant is substantially stationary. Such a dead zone can be created by any suitable means, for example by arranging baffle plates to divert the flow of liquid from the neighborhood of the jets, or by constructing the spinning vessel of suitable shape to produce this effect. The extent of this dead zone may with advantage be such that the materials, by the time they emerge from it into the region of moving liquid, have undergone coagulation to an extent which renders them sufficiently strong to withstand a relatively high tension. When a plurality of spinning orifices is so arranged that the materials extruded converge and thereafter pursue a common path, the transition from the dead zone into the region of movement may be effected beyond or in the neighbourhood of the point of convergence. When the materials are extruded directly into a counter-current of coagulant without passing through any intermediate dead zone, the velocity of the liquid in the neighbourhood of the jet should be such that the materials extruded are not subjected to a greater stretch than they are able to bear. At no point in their path, in fact, should the materials be subjected to a tension approximating to the maximum which they are able to withstand at that point.

The coagulating liquid will not be entirely stationary in the neighbourhood of the spinning jets, or at any point along the path of the materials, since the composition of the coagulant must be maintained throughout the process within the limits necessary to ensure regularity. The movement caused by the continuous or intermittent replenishment of the coagulant may however be very slight.

The materials may, if desired, be extruded into a body of liquid flowing (preferably relatively slowly) in the direction of extrusion, and may then be carried directly through a body of liquid flowing counter to the direction of travel of the materials. The passage of the materials through the counter-current may take place at any stage in the coagulation processes, or the liquid drag may be applied even after coagulation is complete.

The path of the moving body of liquid may be determined by confining it in any suitable manner, as, for example, within the walls of a suitably shaped vessel. Thus for example the liquid may flow through cylindrical, conical or other suitably shaped enclosed vessels, or through suitably shaped open baths or channels. The velocity of the liquid may be uniform, or may vary gradually or abruptly along the path of the materials. Thus for example the cross section or bore of the vessel through which the liquid flows may decrease or increase gradually or abruptly, so as to introduce a corresponding increase or decrease in the velocity of flow over any desired part of the path. Again the velocity at any point in the path may be increased by injecting fresh liquid, or decreased by removing a part of the liquid, in the neighbourhood of that point.

The tension necessary to draw the materials

through the counter-current may be applied in any suitable way, for example by means of positively driven guide rolls or other tensioning devices situated along the path of the materials, or by means of any suitable collecting device.

The invention may be carried out in a very simple way by extruding in an upward or downward direction into a bath or trough containing a body of coagulant substantially stationary in the neighbourhood of the jets, and passing the extruded materials therefrom in an upward direction through a descending body of coagulant. The descending stream of coagulant may flow through a cylindrical or other suitably shaped enclosed vessel or through an open vessel, for example an open channel inclined at a suitable angle, said vessels being suitably supported above the trough into which the coagulant flows. The coagulant may overflow or be drawn off from the bath or trough in any suitable manner. Again, the materials formed by extrusion may pass downwards through a stationary column of liquid supported above the trough by atmospheric pressure, and may be carried through the trough into a column of liquid flowing counter to the direction of the materials. Alternatively the jets may be so situated that the materials pass first into a column of liquid flowing in the direction of extrusion, for example vertically downwards, and are carried by the flow of liquid into the bath, whence they are drawn off through a counter-current of coagulant as previously described.

In order to obtain the advantages of extruding into a body of liquid substantially at rest or moving in the direction of extrusion, and passing the materials therefrom into the counter-current, it is not necessary for the jets to be situated in an open bath or trough as described above. Thus, for example, the jets may be situated in a side tube entering the main vessel, through which the counter current flows, at any angle; the liquid in the side tube may be substantially at rest or may move in the direction of extrusion, so assisting the passage of the materials into the main vessel. Where, for example, the descending column of coagulant flows through the main vessel, which should in this case be enclosed, the side tube in which the jets are situated may be closed at the end, suitable means being provided for replenishing the coagulant, and the liquid therein may be substantially at rest or may flow into the main vessel. On the other hand, the side tube may be open at the top, in which case it must slope downwards into the main vessel. The main vessel and side vessel may have any desired form. They may for example be of circular cross-section, and the cross-section may vary along their length as desired.

The principle of the type of apparatus described above may be applied in various ways. For example the spinning vessel may comprise three limbs in the form of a Y or an inverted Y, the arrangement being such that the counter-current of liquid flows through two of these limbs while the extrusion takes place in the third limb, in which the coagulant may be substantially at rest or may flow in the direction of extrusion. The materials formed in the third limb are drawn out through the limb by which the counter-current of liquid enters. In an alternative form of apparatus open channels constitute the two upper limbs of the Y shaped spinning vessel.

The arrangements described above are such that the materials pass immediately from the

region in which the jets are situated to that in which they are subjected to the drag of a counter-current of coagulating liquid, without leaving the liquid. This is not, however, essential to the process. The jets may for example be situated in the counter-current itself provided the velocity of said current is suitably controlled, so as not to be too great in the neighborhood of the jets. On the other hand the materials may pass out of the body of liquid in which extrusion occurs before entering the counter-current. Thus extrusion may be effected in a suitable vessel in which the coagulating liquid is substantially at rest or is moving with relatively small velocity in the direction of extrusion, and the materials may pass through the air before encountering the stream of coagulant in which the liquid drag is applied. When this arrangement is adopted it is preferable that the air path should be relatively short, and that the conditions should be such that the materials entering the counter-current of the coagulant are in a relatively plastic condition. Any godet or similar device employed in carrying the material from the extrusion bath into the bath in which the liquid drag is applied should not itself exert an excessive tension on the materials. In a simple form of apparatus in which the materials pass through the air before meeting the counter-current of coagulant, extrusion occurs in a cylindrical, conical or other suitably shaped vessel through which a current of coagulant descends relatively slowly. The materials emerge through a small orifice at the bottom of this vessel, and pass over a suitable rotating guide positively driven at such a speed as to exert little or no tension upon the materials, and are then carried in an upward direction through a cylindrical or conical vessel down which a current of liquid is flowing with such velocity as to exert the required drag upon the materials. The materials are withdrawn at the top of this vessel by means of any suitable drawing off device.

The flow of the coagulant may be produced by any suitable means, for example, by the use of a sufficient head of liquid, or by the action of injectors, ejectors or pumps. The arrangements may be such that a proportion of the coagulant circulates continuously through the apparatus, further coagulant being introduced at some point in the circuit, and a corresponding amount of spent coagulant being continuously drawn off.

The velocity of the coagulant at any point in the path of the materials will in general be maintained constant, when once the process has been set in operation. In starting however, it is of advantage to set the liquid throughout the whole system moving in such a direction as to assist the travel of the materials towards the collecting device. Then when the collecting device has commenced to draw off the materials, the velocity of flow in the various parts of the system may be adjusted as finally required. The system should therefore be designed so that variation of the velocity of flow can be simply effected, and in particular so that where during normal working the liquid flows counter to the direction of travel of the materials in starting up said liquid may remain at rest or move in the same direction as the materials. In order to produce particular effects, the velocity of the coagulant may be varied during the normal working of the process. Thus for example filaments and the like of variable denier may be produced by intermittently varying the velocity of flow of the coag-

ulant. This variation may take place at regular or irregular intervals. A similar effect may be produced by varying the velocity of the spinning pumps, as described in U. S. application S. No. 390,400 filed 4th September, 1929.

In carrying out the invention each coagulating bath, funnel or other form of coagulating vessel may be provided with one, two or more jets. These may be multiple jets or, especially in case it is required to spin materials of very heavy denier, e. g. bristles, ribbons and the like, single jets. The jets or dies may be of any suitable form. Where the coagulating vessel is provided with two or more jets it is of advantage to isolate the materials issuing from the respective jets.

Coagulation of the materials may or may not be completed before the materials leave the counter-current of coagulant liquid. It is however of advantage for the materials to retain some degree of plasticity after leaving the coagulating liquid or even throughout the whole of their path to the drawing off device. The materials may pass from the vessel in which the liquid drag is applied into one or more further treatment baths which may or may not contain coagulating or fixing media. A further stretch may if desired be applied in such subsequent treatment baths. In these baths also the liquid may be caused to move in a direction such as to resist the travel of the materials. In applying the counter-current principle to further treatment baths, substantially the same considerations arise as when the principle is applied to the coagulating baths. As in the coagulating baths so in further treatment baths the velocity of the counter-current employed will depend on the viscosity of the treating liquid and the plasticity of the materials. It will usually be advantageous in further treatment baths to employ a liquid of greater viscosity and/or flowing at a higher velocity than the liquid in the coagulating baths. The treating baths may be designed so as to permit of varying the velocity of the moving liquid in the same way as has been indicated in the case of spinning baths.

The process of stretching the materials by drawing them through a current of liquid flowing so as to oppose the travel of the materials, though described above as a step in a wet spinning process, may be applied to filaments, threads, yarns, films and the like continuously with their production by any spinning process. Further the process may be applied to fully formed or finished materials, which may be softened or plasticized with a view to facilitating stretching. The materials may for example be passed through or immersed in a bath containing any suitable swelling agent for the cellulose derivative. I prefer to soften continuously with the drawing of the material through the counter-current. Where a softening agent is employed, the nature of said agent, the length of time during which the material is in contact therewith and the temperature, will naturally be such that a sufficient degree of plasticity is induced in the materials to permit of the stretching operation being effected. In general the time and/or concentration may be decreased with increasing temperature. In applying a softening treatment prior to or as a separate operation from the stretching process, a relatively high concentration of solvent may be obtained on the filaments or other materials by applying a more dilute solution of the solvent in a volatile diluent, and producing the high concentration by evaporation of the diluent. Thus,

for example aqueous solutions of the relatively non-volatile organic liquid reagents referred to below may be allowed to dry in or evaporate on the materials. The concentration of the reagents or the conditions under which they are applied may be varied with the type of material being treated and with its cross-section. When the material passes first through a softening bath and then through the air into the stretching bath, the boiling point of the softening liquid should not be so low that all or substantially all the liquid evaporates from the material before the stretching bath is reached.

The air path between softening and counter-current stretching baths may be very short or may be eliminated altogether, but even in this case it is of advantage to employ softening agents of moderately high boiling point in order to minimize losses due to evaporation.

Any suitable softening agent may be employed. Among those available may be mentioned acetone, methyl ethyl ketone, cyclo hexanone and other ketones, butyl acetate, ethyl lactate, diethyl tartrate, mono- or di-ethers of glycols generally, for example ethylene glycol mono-methyl ether and dioxane, dibutyl tartrate and other aliphatic esters, diacetone alcohol, phenol, thiocyanates, zinc chloride and other inorganic swelling agents. Mixtures of such agents may be employed, for instance mixtures of acetone and diacetone alcohol. The softening agent may be diluted with a non-solvent liquid or diluent, for instance water, benzene or the like. The concentration of the softening agent will depend to some extent on the solvent power of the agent used. By way of example I may mention that diacetone alcohol, diethyl tartrate and ethyl lactate may be employed in aqueous or alcoholic solution in a concentration of above 25%, and in fact very good results are obtained with concentrations of 25 to 65% strength.

Preferably the softening agents employed are such that by reason of their innocuous properties they need not be washed out of the materials and can remain therein to be removed by evaporation only. Good results are obtained with relatively non-volatile neutral organic solvents diluted with a non-solvent diluent, for example aqueous solutions of diacetone alcohol, diethyl tartrate, mono- or di-acetin or ethyl lactate.

The moving liquid in the counter-current stretching baths may be such as to harden the materials. Thus liquids which are non-solvents for the cellulose derivative but solvents for the softening liquid may be employed. It is preferable however to employ for the counter-current liquids having a softening effect. The materials may then undergo a hardening treatment after emergence from the stretching baths. This may for example consist in removing any softening liquid remaining in the materials by means of heat or through the action of a suitable treating liquid. The process may be carried out so that the materials retain some degree of plasticity after emerging from the counter-current stretching baths. Whatever the nature of the liquid employed in the counter-current stretching baths it should preferably have a high viscosity.

The materials may be collected and/or twisted by any suitable means. They may, for example, be collected in a spinning box, or wound on a bobbin, swift or other package. The tension necessary to draw the materials through the counter-current may be applied by the collecting device alone or assisted by any other suitable

means such, for instance, as positively driven guide rolls suitably situated along the path of the materials. When the materials are subjected to a further liquid treatment after leaving the counter-current, the treating liquid may be caused to flow in the direction of travel of the materials, as described in U. S. Patent No. 2,027,419 of January 14, 1936 filed 16th January, 1931 and may in this way supply at least a part of the required tension. The actual tension exerted in this way will of course depend on the velocity of flow and the viscosity of the liquid in the treating bath. Where the treating agent itself does not possess the desired viscosity, suitable substances may be added to the bath to increase the viscosity. When the path of the materials is relatively long it is particularly advantageous for the tension exerted by the collecting device to be supplemented by suitable means along said path. Further, such means may provide the whole of the tension required, the collecting device being driven at such a speed as to exert no tension on the materials.

Before collecting the materials on bobbins, in spinning boxes etc., their plasticity should be sufficiently reduced to prevent coalescence on the package. Preferably substantially the whole of the solvent or swelling agent is removed before collecting.

The materials may be subjected continuously with the stretching process of the invention to any desired after-treatment. They may for example be so treated as to modify their lustre, or to produce metallic or other effects; they may be coloured in fast shades, or a fugitive colouration may be imparted for purposes of identification; they may be weighted, mordanted, saponified or otherwise treated so as to modify their dyeing properties; they may be subjected to sizing or other processes designed to facilitate subsequent textile treatments.

When the process of the invention is applied to the production of the materials by a spinning process, various effects may be obtained by varying the composition of the spinning solution. Finely divided relatively insoluble inorganic or organic substances, for example finely divided barium sulphate or titanium oxide, may be incorporated in the spinning solution according for example to the processes of U. S. applications S. Nos. 444,622 filed 15th April 1930, 464,122 filed 26th June 1930, 473,781 and 473,782 filed 7th August 1930, and 488,829 filed 15th October 1930, or may be formed in the materials by interaction between a soluble salt contained in the spinning solution and a constituent of the coagulating liquid. Thus for example barium sulphate may be formed within the materials by incorporating barium thiocyanate in the spinning solution and spinning into a bath containing sulphuric acid or a sulphate. In this way products of subdued lustre may be obtained. Processes for obtaining such effects are described in U. S. application S. No. 303,602 filed 1st September, 1928.

The spinning solution may contain non-solvents compatible with the solvent mixture, for example paraffin oil, petroleum ether or other hydrocarbons, castor oil or other animal or vegetable oils. Other substances may be incorporated in the spinning solution in order, for example, to facilitate the spinning process or to improve the properties of the product in some particular respect. Where the spinning solution contains non-solvents for the cellulose derivative these should not, of course, be present in sufficient

proportion to precipitate the cellulose derivative.

The process of the invention may be carried out at atmospheric temperature or at temperatures substantially in excess of or below atmospheric temperature, for example temperatures above 20°, 50°, or even higher according to the nature of the materials treated and of the treating (e. g. coagulating) liquid, and the magnitude of the tension applied. In a spinning process, for example, drawing down of the materials is facilitated and products of lower denier may readily be obtained as described in U. S. application S. No. 316,617 filed 1st November 1928, or by maintaining the temperature of the coagulant above the boiling point of a constituent of the spinning solution hollow filaments may be obtained, as is also described in this specification. If desired, a temperature difference may be maintained along the path of the materials in the spinning bath and/or in any subsequent treatment baths.

Various types of apparatus suitable for carrying out the present invention are shown on the annexed drawing, but it is to be clearly understood that they are given only by way of illustration and that the invention is in no way limited thereto.

In the accompanying drawing:

Figs. 1 to 4 show diagrammatic views of apparatus suitable for carrying out the invention in which drag is exerted on the filaments by a liquid medium at least part of which moves countercurrent to the direction of travel of the filaments.

Fig. 5 is a sectional view along the line B—B of Fig. 4.

Fig. 6 shows another form of apparatus in which the coagulating medium in the region of the jet flows in the direction of travel of the filaments.

Fig. 7 is a sectional view along the line A—A of Fig. 6.

Fig. 8 shows a form of apparatus suitable for the treatment of formed filaments or other products.

Fig. 9 shows a form of apparatus suitable for use in the stretching of formed filaments or other products continuously with their production by dry spinning methods.

Fig. 1 shows an apparatus consisting of a cylindrical vessel 10 into which a bundle of filaments 12 is extruded upwards through a spinning jet 11. A coagulating liquid is introduced through a pipe 14 by means of an annular channel which surrounds the upper constricted portion of a funnel 13. This upper portion may be of any desired length, and in order that this may be clear it is shown as broken in each of the illustrations. The bundle of filaments passes upwards through the funnel and meets a counter-current of gradually increasing velocity, until it enters the narrow portion of the funnel where the maximum force of the moving liquid is exerted. It passes up this channel over a guide and is taken up on the roller or bobbin 16. The coagulating medium issues from the vessel through the outlet 15.

Fig. 2 shows a similar form of apparatus but in this case the bundle of filaments 12 extruded from the jet 11 passes through two funnels 13 and 17, the bottom one of which is either positioned close to the base of the vessel 10, or else, as shown in the illustration, supported actually upon the base. In this latter case the bottom of the funnel is formed with openings of a semi-circular or other convenient shape, as is shown in

Figs. 4 and 5, so that a small quantity of the coagulating liquid may flow through this funnel. In this manner the medium surrounding the jet is renewed sufficiently frequently for uniform coagulation of the filaments to be obtained, while at the same time the counter-current in the region of the spinning jet is reduced to a very small amount. As in the previous illustration the liquid medium is introduced through a pipe 14 and flows out at the bottom of the vessel through a pipe 15.

Another form of apparatus is shown in Fig. 3, in which the containing vessel 10 is itself made with its upper part in the form of a funnel. The spinning jet 11 from which the bundle of filaments 12 is extruded is surrounded by a cylindrical partition 19 which serves to protect the filaments, during the first stages of their coagulation, from the full force of the counter-current. The coagulating medium entering through the pipe 14 leaves the vessel through the pipes 15 and 18, and by suitably dimensioning these two outlets the ratio of the volumes of liquid issuing from them may be adjusted to any desired amount. For example, by making the outlet 18 of relatively small dimensions the volume of liquid flowing from the cylinder 19, and consequently the counter-current in the region of the jet, may also be kept small.

Fig. 4 and Fig. 5, a cross-section on the line B—B of Fig. 4, show a form of apparatus which may be employed when it is desired to exert a relatively large tension upon the filament, or when a coagulating medium of relatively small viscosity is employed. In this modification the coagulating medium entering through the pipe 14 passes through an injector device 23 so that a suction is exerted and some of the liquid in the vessel 10 is drawn into the pipe. When this modification is employed, a suitable head of liquid may be maintained above the level of the injector device by controlling the rate of inflow and outflow of the coagulating medium through the pipes 14 and 15. The main body of liquid introduced by the injector device 23 passes down the funnel 13 into the body of the vessel 10 and out through the pipe 15. Concentric with this pipe 15 is a smaller pipe 20 connected by an annular channel with another funnel 17. In consequence of the liquid flow through 15 suction is exerted at the outlet from the pipe 20 so that the liquid is withdrawn from the funnel 17. In this manner a relatively small flow down the upper narrow portion of the funnel 17 is induced, and also a slight upward flow from the bottom of the funnel through the openings 22 in the direction of movement of the bundle of filaments 12. A semi-circular baffle plate 21 is inserted in order to prevent an upward flow towards the outlet 15 causing a turbulent flow in the funnel 17.

Fig. 6 and Fig. 7, a cross-section on the line A—A of Fig. 6, show another form of apparatus in which the coagulating medium in the region of the jet 11 flows in the direction of travel of the filaments. The apparatus consists of a cylindrical vessel 10, the upper part of which has the shape of a funnel. Two inlets for liquid medium are provided, one 14 in the upper constricted portion of the funnel, through which the liquid flows in a downward direction, and the other 25 in the base of the vessel, the liquid introduced at this point flowing upward in the direction of travel of the filaments. In this form of apparatus therefore the bundle of filaments 12 ex-

truded from the jet 11 passes first through a current of liquid moving in the direction of their travel, and afterwards through a counter-current in the upper part of the vessel. The coagulating medium issues from the vessel by the pipe 15, and in order to prevent the outflow from producing a lateral displacement of the bundle of filaments a suitable baffle 24 is provided.

Fig. 8 shows a form of apparatus for the treatment of formed filaments or other products. The filaments 26 are unwound from a bobbin 27 or a swift or other device, and are passed in an upward direction through the opening 15, from which the stretching liquid introduced through the pipe 14 issues. The filaments or other products may have been previously brought to a plastic condition by treatment by immersion in a suitable liquid or by any other suitable method, or a liquid having a plasticizing or softening action on the filaments may be used to produce the stretching force.

Finally, Fig. 9 shows a form of apparatus which may be applied to the stretching of filaments or other products continuously with their production by dry spinning methods. The filaments 36 issuing from the dry-spinning cell 35 are passed round a draw-roller 37 and a back guide 38, over or round a roller 28, where they are treated with a plasticizing medium by means of a spray 29, and then down the vessel 31, through which a suitable liquid medium, introduced by an inlet pipe 14, passes upwards and out through the pipe 15. After issuing from the vessel 31 the filaments pass over a guide 39 and then round a draw-roller 33 which exerts a stretching force. At the same time they are washed by means of the liquid from the spray 32. The vessels 30 and 34 serve to collect and carry off the excess liquid.

What I claim and desire to secure by Letters Patent is:—

1. Process for the manufacture of artificial filaments, threads, ribbons, films and similar materials which comprises extruding a spinning solution containing cellulose acetate into a liquid medium, which, in the neighborhood of the jet, moves in the direction of travel of the materials to form materials having a continuous structure, and withdrawing the materials by means of a suitable drawing device, through a body of liquid moving so as to exert a drag upon the materials.

2. Process for the manufacture of artificial filaments, threads, ribbons, films and similar materials, comprising extruding a spinning solution containing an organic derivative of cellulose into a liquid medium to form materials having a continuous structure, said liquid medium moving counter to the direction of the materials so as to exert a drag thereon, drawing the materials through said liquid medium by means of a drawing roller and conducting the materials to a winding device on which they are wound.

3. Process for the manufacture of artificial filaments, threads, ribbons, films and similar materials, comprising extruding a spinning solution containing cellulose acetate into a liquid medium to form materials having a continuous structure, said liquid medium moving counter to the direction of the materials so as to exert a drag thereon, drawing the materials through said liquid medium by means of a drawing roller and conducting the materials to a winding device on which they are wound.

4. Process for the manufacture of artificial fil-

aments, threads, ribbons, films and similar materials, which comprises extruding a spinning solution containing an organic derivative of cellulose into a liquid medium to form materials having a continuous structure, said liquid medium being in the neighborhood of the jet substantially stationary and in another part of its path moving counter to the direction of the materials so as to exert a drag thereon, drawing the materials through said liquid medium by means of a drawing roller and conducting the materials to a winding device on which they are wound.

5. Process for the manufacture of artificial filaments, threads, ribbons, films and similar materials, which comprises extruding a spinning solution containing cellulose acetate into a liquid medium to form materials having a continuous structure, said liquid medium being in the neighborhood of the jet substantially stationary and in another part of its path moving counter to the direction of the materials so as to exert a drag thereon, drawing the materials through said liquid medium by means of a drawing roller and conducting the materials to a winding device on which they are wound.

6. Process for the manufacture of artificial filaments, threads, ribbons, films and similar materials, comprising extruding a spinning solution containing an organic derivative of cellulose into a coagulating bath containing a substantial proportion of a substance capable of softening the cellulose derivative to form materials having a continuous structure, said coagulating bath mov-

ing counter to the direction of the materials so as to exert a drag thereon, drawing the materials through said coagulating bath by means of a drawing roller and conducting the materials to a winding device on which they are wound.

7. Process for the manufacture of artificial filaments, threads, ribbons, films and similar materials, comprising extruding a spinning solution containing cellulose acetate into a coagulating bath containing a substantial proportion of a substance capable of softening the cellulose acetate to form materials having a continuous structure, said coagulating bath moving counter to the direction of the materials so as to exert a drag thereon, drawing the materials through said coagulating bath by means of a drawing roller and conducting the materials to a winding device on which they are wound.

8. Process for the manufacture of artificial filaments, threads, ribbons, films and similar materials, which comprises extruding a spinning solution containing cellulose acetate into an aqueous medium containing a substantial proportion of a softening agent for cellulose acetate so as to form materials having a continuous structure, said medium moving counter to the direction of the materials so as to exert a drag thereon and drawing the materials through said medium by means of a drawing roller and conducting the materials to a winding device on which they are wound.

HENRY DREYFUS.

CERTIFICATE OF CORRECTION.

Patent No. 2,054,852.

September 22, 1936.

HENRY DREYFUS.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 5, first column, line 8, strike out the words "filed 16th January, 1931"; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 3rd day of November, A. D. 1936.

Henry Van Arsdale
Acting Commissioner of Patents.

(Seal)