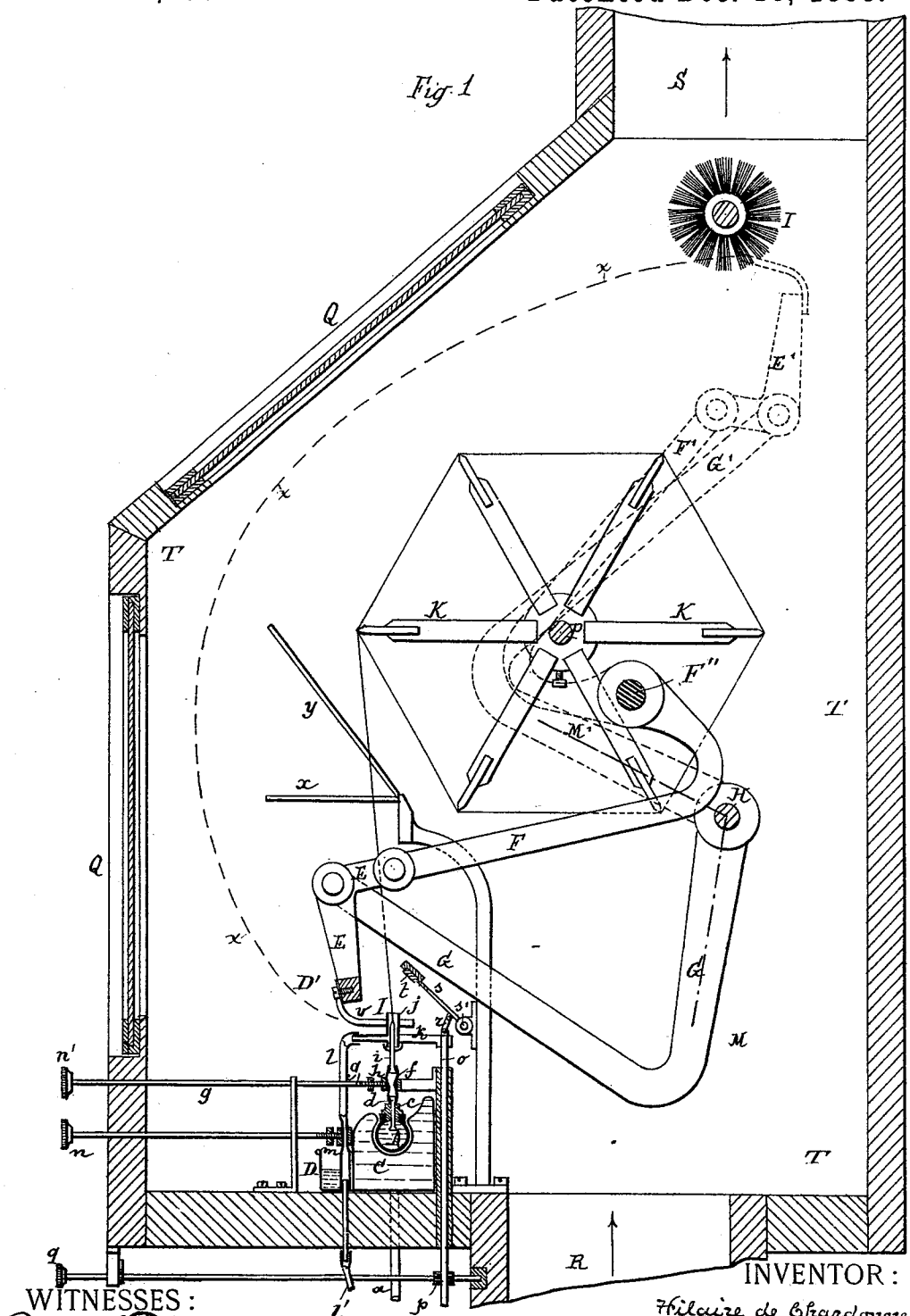
(No Model.) 4 Sheets—Sheet 1.

H. DE CHARDONNET.

ARTIFICIAL SILK AND METHOD OF AND APPARATUS FOR MAKING THE SAME.

No. 394,559. Patented Dec. 18, 1888.

Fig. 1



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ARTIFICIAL SILK AND METHOD OF AND APPARATUS FOR MAKING THE SAME.

No. 394,559. Patented Dec. 18, 1888.

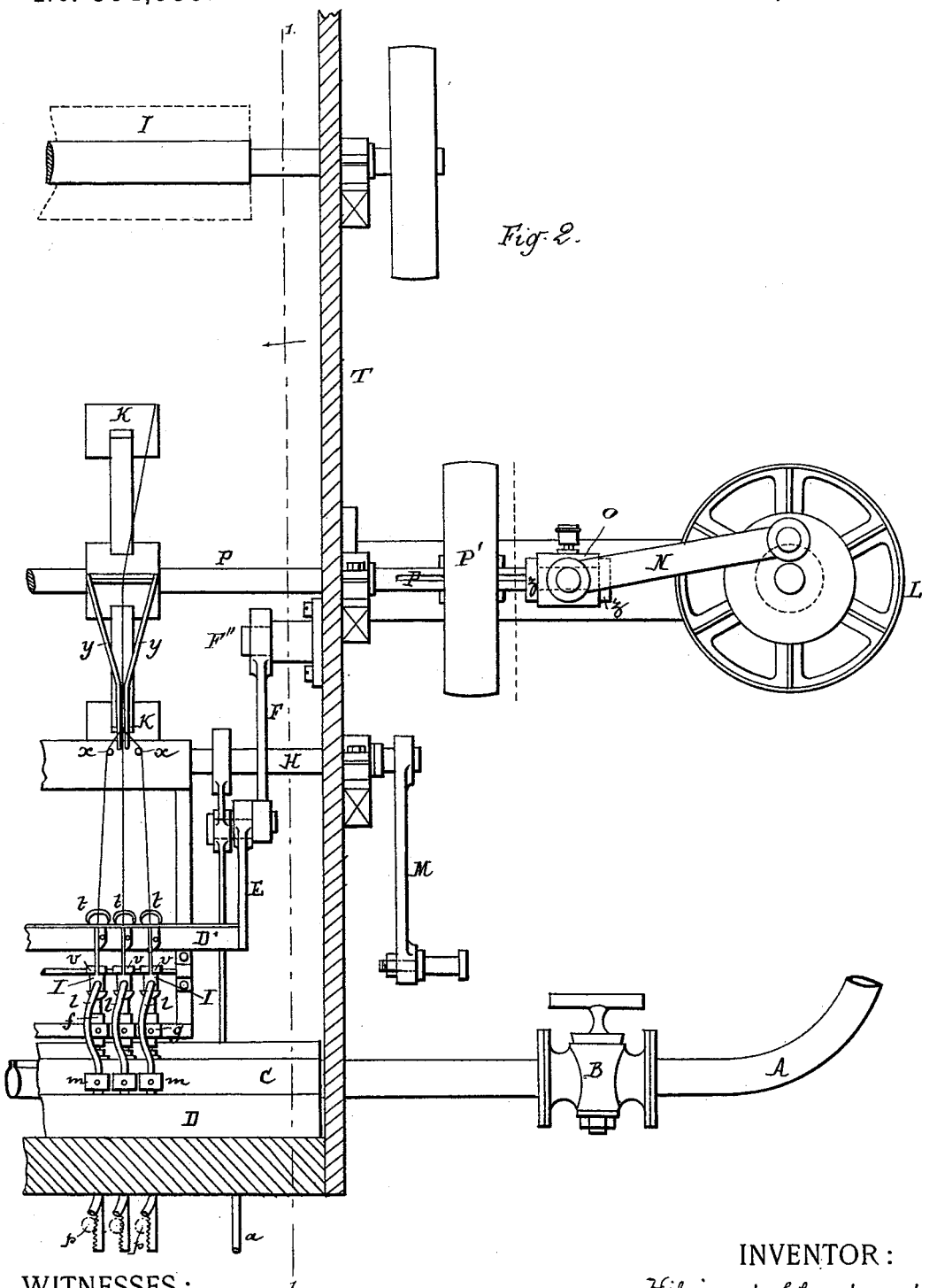

Fig. 2.

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ARTIFICIAL SILK AND METHOD OF AND APPARATUS FOR MAKING THE SAME.

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Fig. 5.

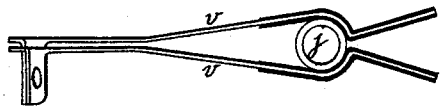


Fig. 6

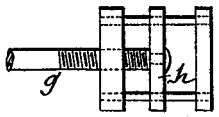


Fig. 4

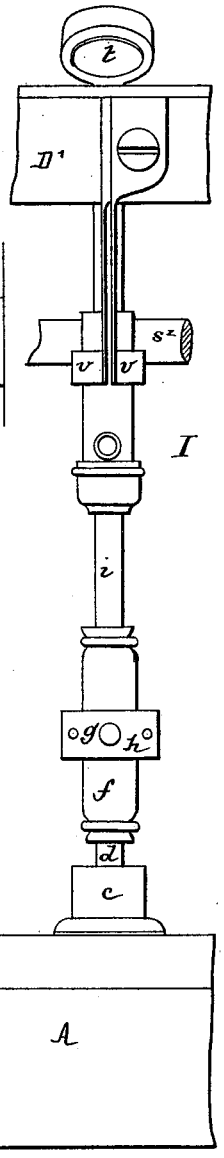
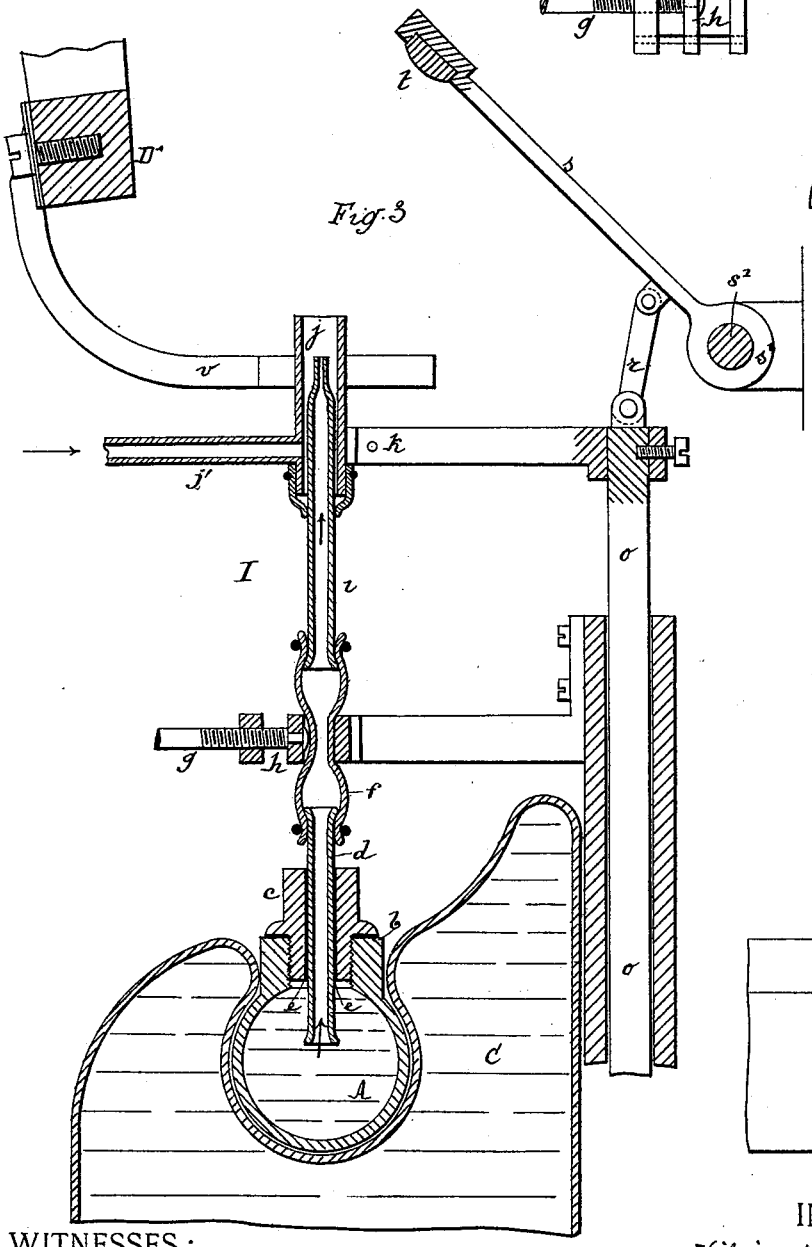


Fig. 3



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ARTIFICIAL SILK AND METHOD OF AND APPARATUS FOR MAKING THE SAME.

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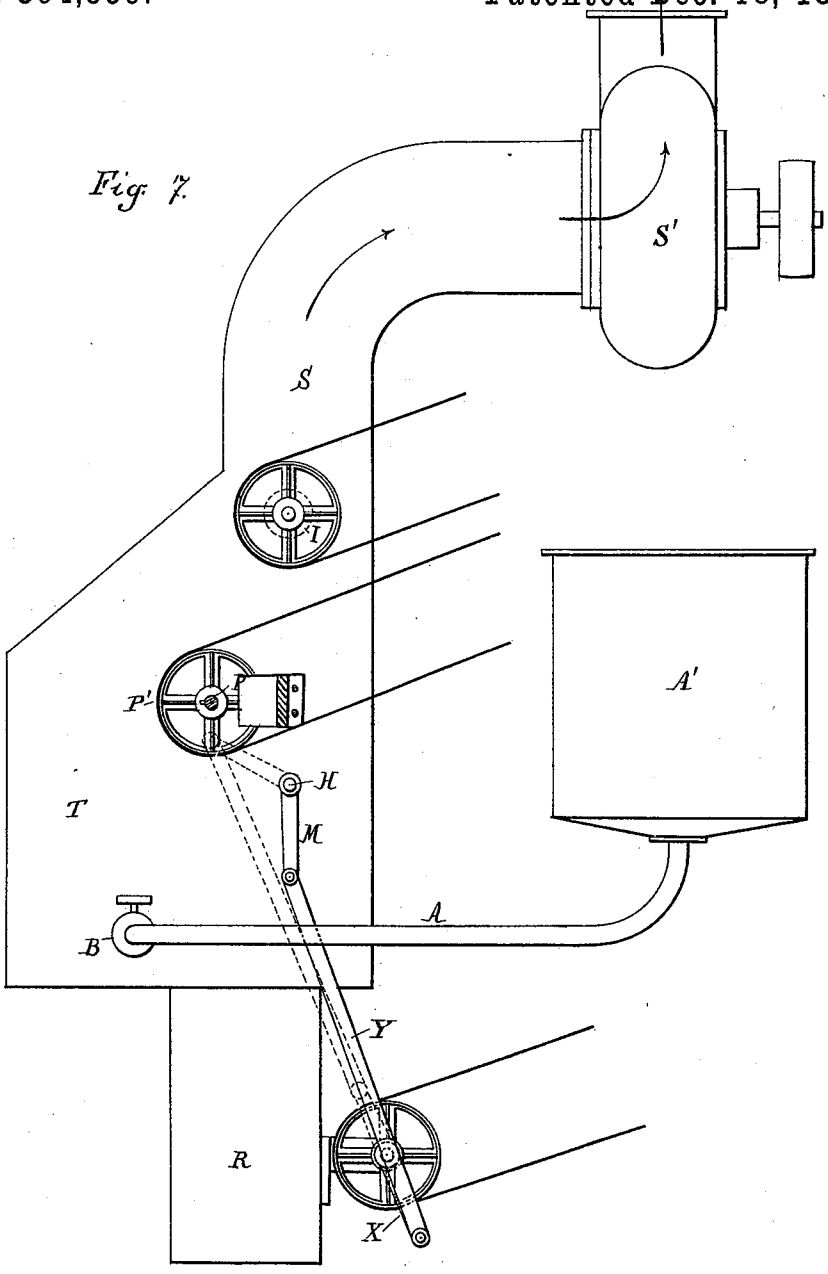
Fig. 7.



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UNITED STATES PATENT OFFICE.

HILAIRE DE CHARDONNET, OF BESANÇON, DOUBS, FRANCE.

ARTIFICIAL SILK AND METHOD OF AND APPARATUS FOR MAKING THE SAME.

SPECIFICATION forming part of Letters Patent No. 394,559, dated December 18, 1888.

Application filed December 20, 1886. Serial No. 222,033. (No model.) Patented in France November 17, 1884, No. 165,349, and November 13, 1885, No. 172,207; in Italy January 23, 1885, XVIII, 17,844, XXXV, 265; in Belgium May 16, 1885, No. 68,890, and February 8, 1887, No. 76,272; in Spain July 23, 1885, No. 7,849; in Austria-Hungary December 18, 1885, No. 31,310; in Germany December 20, 1885, No. 38,368, and in England February 15, 1886, No. 2,210.

To all whom it may concern:

Be it known that I, LE COMTE HILAIRE DE CHARDONNET, a citizen of the French Republic, residing at Besançon, Doubs, France, have invented a new and useful Improvement in Artificial Silk and Method of and Apparatus for Making the Same, of which the following is a specification.

This invention is the subject of the following foreign Letters Patent: France, No. 165,349, dated November 17, 1884, and certificates of addition thereto dated December 23, 1884, and May 7, 1885; France, No. 172,207, dated November 13, 1885, and certificate of addition thereto dated December 31, 1886; Italy, Registro Generale, Volume XVIII, No. 17,844, Registro Attestati, Volume XXXV, No. 265, dated January 23, 1885, and certificate of addition thereto dated January 5, 1887; Spain, No. 7,849, dated July 23, 1885, and certificate of addition thereto dated January 20, 1886; Belgium, No. 68,890, dated May 16, 1885, and a patent of improvement thereon, No. 76,272, dated February 8, 1887; Great Britain, No. 2,210, dated February 15, 1886; Austria-Hungary, No. 31,310, dated December 18, 1885, and Germany, No. 38,368, dated December 20, 1885.

My invention relates to the production of artificial silk-like filaments from viscous liquids. The threads or filaments produced according to my invention are supple, glossy, and have sufficient strength to enable them to be used for the manufacture of fabrics in the same manner as silk fibers.

According to my invention I draw out the fibers or filaments from a liquid of special composition, being a pyroxyline compound, and form them into threads by means of a special apparatus.

In order that my invention may be well understood, I will proceed to describe in detail the method and the apparatus that I have devised.

I. *Preparation of the liquid.*—The liquid that I employ is a non-structural pyroxyline compound, being a kind of collodion, obtained by dissolving in a mixture of alcohol and ether a quantity of pyroxyline, a metallic protochloride reducing agent, and a small quantity of an oxidizable organic base. The pyroxyline that I employ is obtained in the ordinary manner by subjecting purified cellulose, obtained from the chemical treatment of wood, straw, cotton, rags, or unsized paper or other similar materials, to the action of nitric acid.

In preparing the liquid pyroxyline compound for my artificial fiber I take one hundred grams of pyroxyline, ten to twenty grams of a metallic protochloride reducer, (of iron, chlorine, manganese, or tin,) and 0.20 grams of an oxidizable organic base—such as quinine, aniline, rosaniline, &c.— and I dissolve them in from two to five liters of a mixture of forty per cent. of ether and sixty per cent. of alcohol. I add any suitable coloring-matter which is soluble in this mixture. In dissolving the pyroxyline its fibrous or cellular structure is destroyed, and a non-structural, homogeneous, or amorphous solution is obtained. In order to prepare the liquid in the best condition I find it preferable to dissolve the pyroxyline in the largest part of the mixture of alcohol and ether, and in the rest of the liquid to dissolve the protochloride, the organic base, and the coloring-matter, and subsequently to mix together the two solutions.

The proportions of the ingredients employed will vary according to the nature of the pyroxyline that is used and according to the elasticity and strength that one desires to realize in the thread or filaments that will be manufactured from the liquid.

II. *Spinning the filaments.*—Having prepared the viscous liquid in the manner already described, or any analogous and suitable liquid, I proceed to draw from it the threads or filaments of artificial silk. This is done by causing the liquid to flow out through a minute orifice in such manner that the small stream or filament of liquid as it emerges therefrom enters into a cold liquid of any suitable kind—such as water, for example—so that it is immediately solidified on its exterior. The filament then presents the form of a tube or sheath solid on its exterior and inclosing a central column of liquid which

has not yet solidified. The filament is then drawn or spun out and emerges from the cooling-liquid and is dried by contact with the air, so that it becomes solidified throughout its entire substance. To facilitate the drying and solidification of the filament, I cause a circulation of warm air through an inclosed space or chamber, in which the filaments are reeled.

III. *The apparatus.*—In order to realize practically the spinning of fibers of artificial silk by my method, I have devised a machine for this purpose which is illustrated in the accompanying drawings.

Figure 1 is a transverse section of the apparatus. Fig. 2 is a front elevation of a portion of the apparatus, the box or casing of the spinning apparatus being in vertical section. Fig. 3 is an enlarged fragmentary section answering to Fig. 1 and showing the spinning devices. Fig. 4 is a fragmentary front elevation of the parts shown in Fig. 3. Fig. 5 is a plan of the nippers *v v*, shown in Fig. 3 on the same enlarged scale. Fig. 6 is a fragmentary plan view of the clamp *h* shown in Figs. 3 and 4. Fig. 7 is a side elevation of the apparatus.

The collodion solution, having been well filtered, is placed in a closed vessel or receptacle, A′. (Shown in Fig. 7.) This vessel is lined or coated interiorly with tin, and from it leads a pipe, A, which passes down toward the lower part of the machine and enters horizontally into the same, as shown in Fig. 2. This pipe is provided with a valve or stop-cock, B, to regulate the flow. The closed vessel or reservoir is furnished with an air-pump, (not shown,) which maintains in it a pressure of from two to three atmospheres, in order that the collodion liquid may be expelled with sufficient force to effect the spinning of the filaments. The pipe A extends through the entire length of the machine, wherein it is inclosed or partly surrounded by a water-jacket, C, in the manner best shown in Figs. 1 and 3. In this water-jacket, the walls of which are of thin metal, cold water is caused to circulate. The pipe *a* in Figs. 1 and 2 is that by which the cold water escapes. The pipe A carries all the vertical spinners or nozzles through which the liquid is forced out to form the filaments. These spinners or nozzles I I are arranged in a row equidistantly along the top of the pipe A. Each spinner is made up of several parts, as best shown in Fig. 3. On the top of the pipe A is a thickened boss, *b*, in which is formed a threaded hole, and into this is screwed a socket, *c*, in which is fixed a vertical tube, *d*, a thin packing, *e*, of rubber, vulcanized or not, being interposed around the tube *d* to make a tight joint.

To the upper end of the tube *d* is connected a flexible tube, *f*, of soft india-rubber or other impermeable material which cannot be attacked by the solution, and to the upper end of this flexible tube is connected a tube, *i*, of glass or other suitable material, the upper end of which is reduced to form a contracted nozzle or nipple. Around this nozzle is arranged a glass tube, *j*, which is held by a clamp, *k*, attached rigidly to a vertical slide, *o*. This slide *o* extends downwardly and passes out beneath the inclosing-case of the apparatus, and its lower portion, which is toothed, is engaged by a pinion, *p*, which is turned by a knob, *q*, at the front of the apparatus. By turning this knob the slide *o*, clamp *k*, and surrounding tube *j* are raised or lowered, while the nozzle-tube *i* remains at the same height. Cold water enters by a tube, *l*′, Fig. 1, and is conducted by a flexible rubber tube, *l*, and the lateral branch *j*′ into the interior of the tube *j* and around the nozzle-tube *i*. The water that thus flows in ascends around the nozzle-tube, runs over the top of the tube *j*, and runs down, being finally caught in a trough or gutter, D, Fig. 1. The flow of water is controlled by means of a screw-clamp, *m*, which pinches more or less the rubber tube *l*, and thus more or less reduces the area of the tube, and consequently the size of the stream that passes through it. The clamp *m* is operated from the outside by means of a knob, *n*, at the front of the apparatus. In like manner the flow of the collodion liquid through the flexible tube *f* is regulated by the clamp *h*, which is adjusted by a screw, *g*, which extends out to the front of the machine, and is provided with a knob, *n*′. Fig. 6 illustrates this clamp in detail. Its construction is identical with the clamp *m*. Ordinarily the inclosing-tube *j* extends higher than the nozzle-tube *i*, as shown in Fig. 3, so that the collodion liquid on being forced out through the nozzle enters a body of cold water held in the tube *j*, and is caused to pass through this water until it reaches the level of the top of the tube *j*; but when the machine is not in use it is desirable to close the nozzle, and it is also desirable that there shall be no water above it which might enter it. For this purpose the slide *o* is lowered by turning the knob *q*, thereby lowering the tube *j* until the level of the water therein is beneath the orifice of the nozzle. The same movement acts, by means of a link, *r*, to draw down an arm, *s*, pivoted at *s*′, the extremity of which carries a stopper or pad, *t*, which on being thus lowered closes the said orifice in the nozzle and prevents the solidification of the solution in the capillary tube by the action of the surrounding air or by the water. The flow of water is shut off at the same time by turning the knob *n*.

In the operation of the machine, the parts being in the position shown in Fig. 3, the minute stream of collodion liquid on emerging from the nozzle (the orifice in which should be reduced to a diameter of about one-tenth of a millimeter) is carried up through the tube *j* by the ascending stream of water, being solidified at the same time, and its end hangs or falls over the outside of the tube *j*. The loose end of the filament thus formed must then be grasped and carried to the reel

in order that the spinning operation may commence. To accomplish this result, I provide the mechanism which I will now describe. The reel (shown at K in Figs. 1 and 2) is arranged in the upper part of the apparatus, with its winding-on side over the row of spinners or nozzles I I. A long bar, D′, extends longitudinally in front of and a little above the row of nozzles and is fixed at each end to an elbow-lever, E. This bar D′ carries a series of nippers or grasping-fingers, *v v*, one to each nozzle. These nippers are best shown in Figs. 3, 4, and 5. Each nipper is formed of two elastic springs curved as shown in side view in Fig. 3 and in plan in Fig. 5, and adapted to embrace the inclosing-tube *j* of the nozzle. Both of the springs are fastened to the bar D′ by one screw. The end portions of the springs are coated with soft india-rubber, not vulcanized. In Fig. 3 the nippers *v* are shown as engaging the tube *j*. If the bar D′ were to be moved to the left in this figure, the springs would yield and the nippers would free themselves from the tube, at the same time engaging the overhanging end of the collodion filament. The bar D′ is given such a movement that the nippers *d* travel first to the left and then, having freed themselves from the tube *j* and grasped the filament, sweep upwardly and over the top of the reel K in the path indicated by the dotted line *x x* in Fig. 1, thereby carrying the end of the filament up over the reel and enabling the reel to engage the filament and continue thereafter to draw it out. The speed of movement of the bar D′ in its ascent and the speed of rotation of the reel K are so proportioned to the rate of discharge of the collodion from the nozzle that they act to draw out the filament to the proper degree of attenuation. In order to free the nippers from the filament after they have carried it over the reel, I provide a revolving brush, J, the bristles of which encounter the nippers when the bar D′ reaches its extreme upper position. By this means the nippers are cleaned. The bar D′ is moved up and back two or three times per minute. At the completion of its downward movement its nippers *v v* spring over and embrace the glass tubes *j j*. During the upward movement of the bar its nippers carry the filament between horizontal guide-pins *x x*, which are arranged in a series corresponding to the number of nozzles, and the function of which is to separate the filaments emerging from the respective nozzles. Above the guide-pins *x x* there is arranged a series of inclined guide-pins, *y y*, as shown in Fig. 1, which are arranged in pairs, with the two pins of a pair converging, as shown in Fig. 2. The effect of this arrangement is that as the nippers carry the filaments up the latter are first caught between the pins *x x* and thereby held distinct and parallel and the group of three filaments enters between the extremities of one pair of pins *y y*, and in being drawn down to the parallel portion thereof the three filaments are united to form one thread, which is then wound upon the reel.

The peculiar movement of the bar D′, which I have described, is imparted to it by a special combination of levers. The elbow-lever E at each end of the bar D′ is fulcrumed or pivoted to the end of a bent lever or crank, G, which is fixed on a shaft, H, and to the outer end of this shaft is fixed a crank, M, which is oscillated by hand or by any suitable mechanical connection—such as by a crank, X, and pitman Y, Fig. 7—from the position denoted at M in Fig. 1 to that shown at M′. This oscillatory movement carries the lever G up to the position shown in dotted lines at G′ in Fig. 1, and lifts the elbow-lever E to the position shown in dotted lines at E′. During this movement the lever E is caused to swing outwardly by means of an arm, F, to which its other end is pivoted, and which arm is pivoted at F″ at a point above and in front of the axis of the shaft H. The levers F and G are duplicated at opposite ends of the machine. At the commencement of the operation of the machine a filament will be forced out of reach of the nozzles I I, of which there may be any number, and will flow over the top of the tube *j* of each nozzle. At the first movement of the bar D′, carrying the nippers, all of the filaments, or very nearly all, will be grasped by the nippers and elevated over the reel, and will then be engaged by the reel, after which they will be drawn or spun out without interruption. At the second movement of the bar D′ the nippers will engage any filaments that may have been missed at the first movement, and will add them to the reel. Thereafter, although the movement of the bar D′ continues, it is without effect so long as no interruption occurs in the spinning of the filaments; but whenever a filament breaks the nippers *v*, which engage the nozzle corresponding to that filament, will grasp the overhanging end of the severed filament and carry it up over the reel again, whereupon the interrupted filament is reunited by adhesion with the others of its group. At each ascending movement of the bar D′ the nippers are cleaned by the brush J.

In order that the successive turns of the thread on the reel shall not be wound upon themselves, I provide for causing them to cross angularly from side to side, thereby facilitating the drying of the threads. This is accomplished by imparting to the reel, in addition to its rotary movement, a longitudinal reciprocatory movement by means of the device shown in Fig. 2. A pulley, L, driven at suitable speed, carries a crank, which is connected to a pitman, N, which in turn is coupled to an oiler-box, O. This box is mounted on the shaft P of the reel, being confined thereon between collars *z z*, so that the shaft is free to turn within the box; but as the box is moved endwise the shaft is carried with it. The shaft P may be rotated by a belt passing over a pulley, P′, or by any other means. The

pulley P′ is connected with the shaft by a key and groove, so that the shaft can slide freely and still be rotated by the pulley. The pulley is prevented from sliding with the shaft by means of fingers which embrace its rim, the fingers being supported by the bracket which supports the pulley L. (See Figs. 2 and 7.)

The entire apparatus thus far described, with the exception of the regulating-knobs, is inclosed in a suitable tight box or casing, T, provided with glazed openings Q Q, through which the operator may look in order to observe the operation of the machine. These may be opened in order to regulate or clean the apparatus. A current of air is caused to circulate through the box T, entering at R and passing out at S. For this purpose a fan or blower, S′, Fig. 7, may be provided. This air, which should be heated to about 30° centigrade, (85° Fahrenheit,) serves to dry the threads and carries away with it the spirituous and aqueous vapors which are evaporated within the casing. These vapors are subsequently recovered by passing the air through water or through a refrigerating-machine. The air is then reheated and reintroduced at R. It thus circulates continuously. The alcohol and ether are separated from the water by rectification or by fractional distillation, and serve, without being separated from each other, for dissolving the pyroxyline for forming the collodion solution.

One may, if necessary, accelerate the drying of the threads by forming a partial vacuum in the casing. If this be done, the casing must be made air-tight and all the mechanical parts which communicate with the exterior must be provided with suitable packings.

The new textile provided by my invention may be rendered incombustible by impregnating it either in skeins or, after weaving, with any non-inflammable preparation that may be desired.

I am well aware that filaments bearing some resemblance to silk have been made by spinning glass. A rod of glass is rendered ductile by heating it in a blow-pipe flame, and a fine thread is drawn from it and attached to the circumference of a rapidly-revolving wheel, by means of which a continuous and attenuated filament is drawn out. The fragile and brittle character of spun glass unfits it for use as a substitute for silk and distinguishes it from the product of my invention.

I am also aware that coarse elastic threads suitable for some textile uses, but having no resemblance whatever to silk, have been made from gutta-percha. This gum in a soft condition is forced through a heated die, thereby converting it into a thread, which is then cooled by being passed through water, and is then wound on a reel. It is subsequently rewound onto another reel and stretched somewhat during this operation, whereby it is drawn down to a somewhat smaller diameter.

The filaments produced by my invention are distinguished from those resulting from both these methods by their silk-like quality, being not only glossy and resembling silk fiber in their appearance, but also being so supple, strong, and durable that they form a practicable and valuable substitute for real silk.

I claim as my invention the following defined novel features and combinations, substantially as hereinbefore specified, namely:

1. The new textile material or artificial silk herein described, consisting of filaments of a non-structural pyroxyline compound.

2. The new textile material or artificial silk herein described, consisting of minute filaments of a non-structural pyroxyline compound, combined to form a thread.

3. The new textile material or artificial silk herein described, consisting of filaments of collodion.

4. The improved method of making artificial silk, which consists in preparing a non-structural pyroxyline compound in the condition of a viscous liquid, forcing it out through a minute orifice, and drying the filament thus formed.

5. The improved method of making artificial silk, which consists in forcing a viscous liquid pyroxyline compound through a minute orifice, solidifying the filament thus formed by passing it through a cold medium, and simultaneously drawing out the filament to the requisite attenuation.

6. The improved method of making artificial silk, which consists in forcing a viscous liquid pyroxyline compound through a minute orifice into a body of cold liquid, whereby the exterior of the filament thus formed is solidified, and then drawing the filament from said liquid and through the air, whereby it is dried.

7. The improved method of making artificial silk, which consists in forcing a liquid pyroxyline compound through two or more minute orifices, thereby producing two or more filaments, and simultaneously drawing out these filaments to reduce them to the desired attenuation and combining them into one thread.

8. The improved method of making artificial silk, which consists in dissolving in a mixture of ether and alcohol a quantity of pyroxyline, a metallic protochloride, and an oxidizable organic base, and subsequently forcing this solution through a minute orifice, whereby it is converted into a filament.

9. The improved composition for making artificial silk, which consists of a mixture of pyroxyline, a metallic protochloride, and an oxidizable organic base dissolved in a mixture of ether and alcohol.

10. The combination of the pipe for conducting the liquid from which the fibers are to be made, a series of nozzles branching from said pipe, a reel for winding the filaments drawn from said nozzles, and regulating devices applied to said nozzles for controlling the flow of liquid therethrough.

11. The combination of a pipe for conducting the material from which the fibers are to be made, a nozzle branching from said pipe, a tube inclosing the end of said nozzle, and means for conducting water to said tube, whereby the orifice of the nozzle is submerged in the water contained in said tube.

12. The combination of a pipe for conducting the material from which the fibers are to be made, a nozzle branching from said pipe, a tube inclosing the end of said nozzle, a rubber tube for conducting water to said inclosing-tube, and a clamp applied to said rubber tube and adapted to control the flow of water therethrough.

13. The combination of a pipe for conducting the liquid from which the fibers are to be made, a nozzle branching therefrom, an inclosing-tube arranged over the end of said nozzle and capable of moving up or down thereon, and mechanism for raising or lowering said inclosing-tube.

14. The combination of a pipe for conducting the liquid from which the fibers are to be made, a nozzle branching from said pipe, a tube inclosing the end of said nozzle, means for conducting water to said tube, means for raising and lowering said tube on said nozzle, and a stopper for closing the orifice in said nozzle.

15. The combination of a pipe for conducting the liquid from which the fibers are to be made, a series of nozzles branching from said pipe, a series of tubes, each inclosing the end of one of said nozzles, means for conducting water to said tubes, and independent means of regulating the flow of water to each of said tubes and for adjusting each of said tubes up or down.

16. The combination of the nozzle through which the liquid from which the fibers are to be made is caused to flow, a reel for winding the filament emerging from said nozzle, and a lifting device consisting of nippers adapted to engage the filament and mechanism for carrying said nippers up over said reel.

17. The combination of a nozzle, a reel for winding the filament drawn therefrom, and a lifting device for drawing out the filament and carrying it over the reel, consisting of elastic nippers for engaging the filament as it comes from said nozzle, and mechanism adapted to elevate said fingers beyond the reel.

18. The combination of a pipe for conducting the material from which the fibers are to be made, a series of nozzles branching from said pipe, a reel for winding the filaments drawn from said nozzles, a series of elastic nippers, each engaging one of said nozzles, a lifting-bar carrying said nippers, and lifting mechanism, substantially as described, for moving said bar first laterally until the nippers are disengaged from the nozzles and then upwardly to carry the filaments over said reel.

19. The combination of a nozzle, a reel for winding the filament drawn therefrom, an elastic holding device for engaging the filament as it comes from said nozzle, mechanism consisting of crank-arms connected to said holding device and adapted to elevate it and thereby to draw out said filament and carry it over the reel, and a revolving brush adapted to clean said holding device after the latter has been lifted.

20. The combination, with the nozzles, of the nippers *v v*, the lifting-bar D′, the elbow-levers E, the cranks G, the shaft H, and the rods F.

21. The combination, with the nozzle, of its inclosing-tube *j*, the vertical slide *o*, the pivoted arm *s*, connected to said slide, and the stopper *t*, carried by said arm.

22. The combination of a series of nozzles, a reel for winding the filaments drawn therefrom, a series of guide-pins corresponding to the nozzles for separating the filaments drawn therefrom, and a series of converging guide-pins adapted to unite a plurality of filaments into a single thread before the latter is wound upon the reel.

23. The combination, with the series of nozzles and the reel for winding the filaments drawn therefrom, of a mechanism for imparting a longitudinal reciprocatory movement to said reel during its revolution.

24. The combination, with the spinning and reeling apparatus for making artificial fibers, of a casing inclosing said apparatus and means for causing a circulation of warm air through said casing for effecting the drying of the spun filaments.

25. The method of manufacturing threads from viscous, pasty, or glutinous matters, which consists in projecting said matters while out of contact with air through a small orifice into a fluid medium other than air, so as to prevent the material of which the thread is composed from clogging the orifice and to effect its coagulation as fast as it is protruded.

In witness whereof I have hereunto signed my name in the presence of two subscribing witnesses.

HILAIRE DE CHARDONNET.

Witnesses:
JOS. W. HARPER,
EMIL HENZEL.