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METHOD OF SPINNING COLLAGEN FILAMENTS

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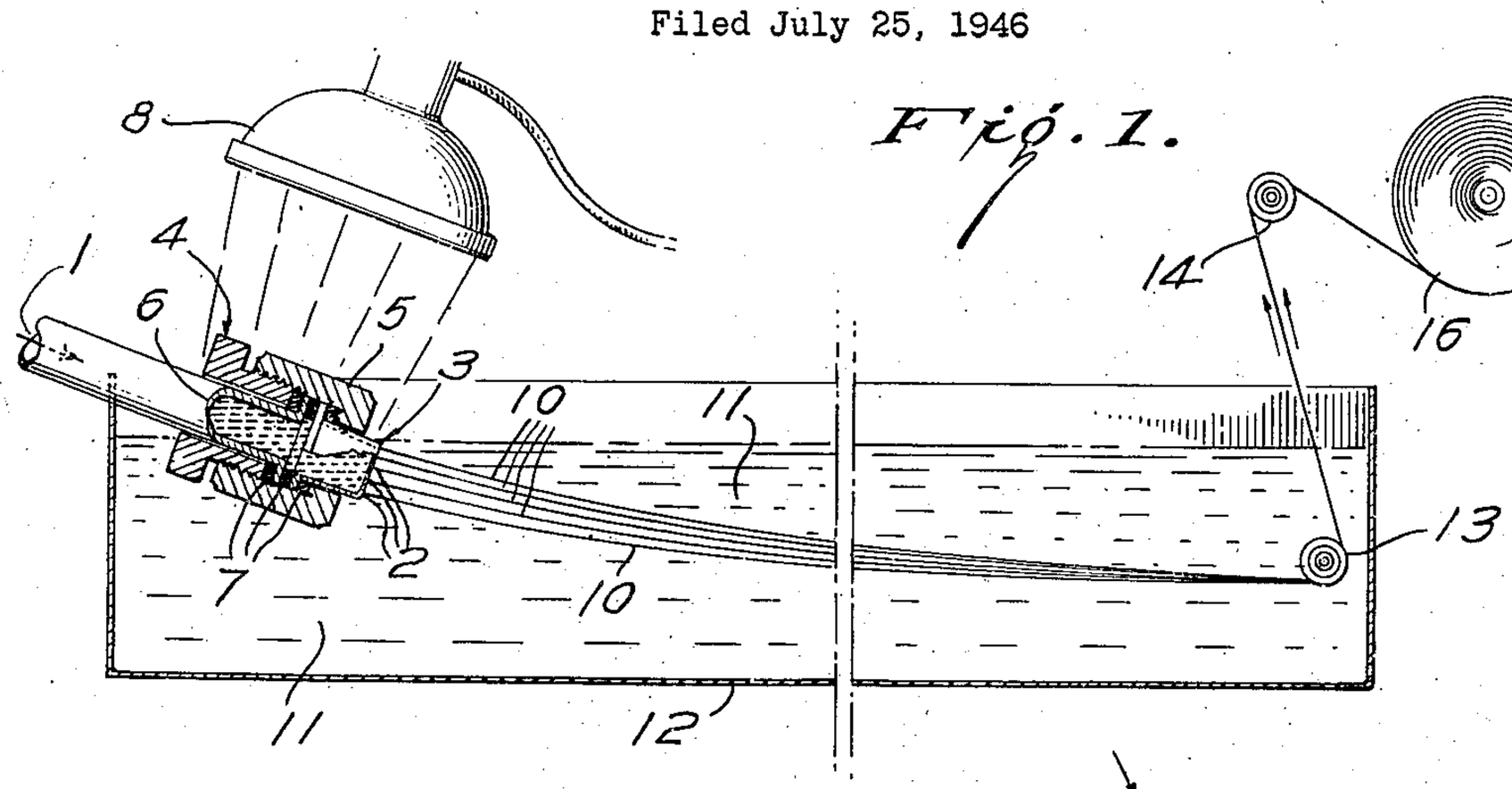

Fig. 1.

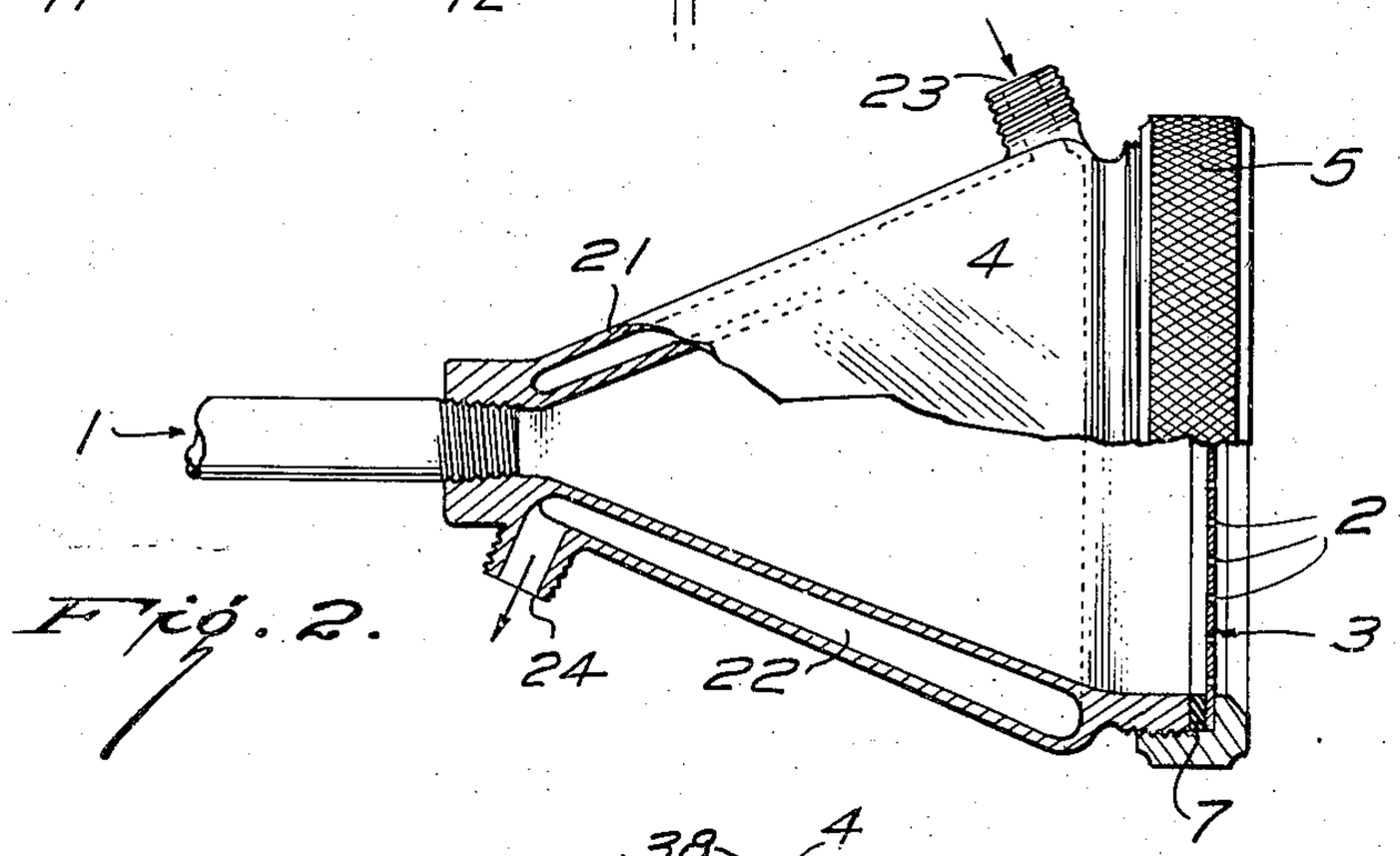

Fig. 2.

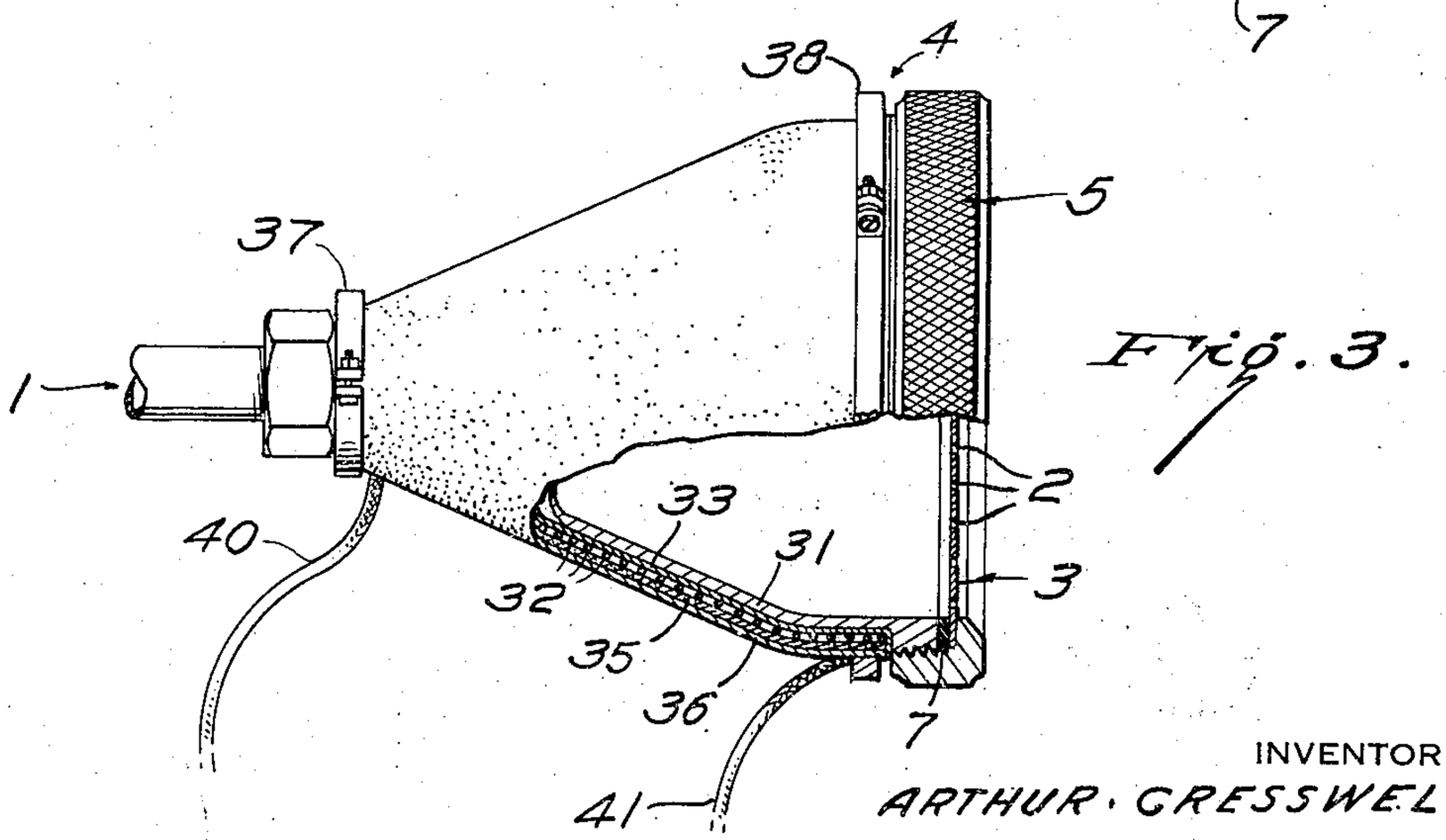

Fig. 3.

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METHOD OF SPINNING COLLAGEN FILAMENTS

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2 Claims. (Cl. 18—54)

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The present invention relates to an improved method for spinning collagen filaments, threads and the like.

An object of the present invention is to prevent the breakage of such collagen filaments, threads and the like in the course of spinning.

Another object of the present invention is to provide multi-filament collagen threads containing substantially no broken filaments and therefore devoid of any frayed appearance.

Other objects of the invention will be apparent from the discussion contained hereinafter.

Heretofore, collagen filaments, threads and the like have been prepared by extruding a solution of collagen into a spinning or coagulating bath. To facilitate such extrusion it has been the practice to maintain such collagen solutions above their respective gelling points, while in order to bring about the most effective coagulation the spinning bath has been maintained at the lowest practicable temperature. The gelling point for collagen solutions of 5–15% is of the order of 35° C., whereas the lowest practicable temperature at which the spinning bath can be maintained is 20° C., in view of the high salt content usually employed. Such spinning has been found to be attended by appreciable breakage of the filaments and in the case of spinning a single filament or thread this results in the breakdown of production and necessitates rethreading the bobbins, rewinding the take-up spool and the like with considerable loss of time. In the case where several filaments are extruded simultaneously in the preparation of a multi-filament thread, breakage of the filaments has not been found to be sufficiently severe to disrupt production but it nevertheless manifests itself in the frayed appearance of the final product.

It has now been found that heating the spinnerette or the coupling in which such spinnerette is held, which is hereinafter referred to as the spinnerette head, or other device through which the collagen solution is extruded for the purpose of spinning, serves to reduce if not substantially eliminate the aforementioned breakage. While heating the spinnerette or spinnerette head to any extent has been found to reduce the amount of breakage sustained, heating sufficiently to raise the temperature of the spinnerette head and the collagen solution contained therein above the gelling point of the collagen solution being spun will minimize such filament breakage. Also, it has been found that heating to raise the temperature appreciably above this point does not effect any additional improvement. For this reason it

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is believed, although it is not known for certain, that the breakage is caused by minute gels plugging the orifices of the spinnerette with resultant disruption of the filament formation. While this may be so, such disruption is only momentary and a new filament is shortly forthcoming through the orifice. There is never complete plugging, even when a single-orifice spinnerette is employed, the effect being simply that the filament is broken.

The spinnerette head may be heated by any suitable method as by jacketing it and employing steam, hot water, hot air, or other fluid media, or by wrapping the head with suitably covered wiring for electrical heating, or by focusing a lamp such as an infra-red lamp upon the spinnerette or the spinnerette head.

The spinnerette containing the orifices is a plate or disc member which is usually flanged, such flanging being frequently so extreme as to result in a hat-shaped member. The spinnerette is mounted in a suitable coupling, the entire piece of apparatus being the above-mentioned spinnerette head. Such plate and coupling may be constructed of any suitable material, preferably metallic, the plate usually being of precious metal.

Figure 1 is a diagrammatic showing of the heating means with relation to the spinnerette head as employed in actual spinning, the heating means being the preferred infra-red lamp.

Figure 2 shows an arrangement suitable for heating by jacketing the head and using hot water, hot air, steam or other fluid media.

Figure 3 shows an arrangement suitable for heating by electrical means.

In the practice of the invention, a collagen solution is prepared by treating the cleaned corium of hide or skin material with a solution of organic acid, preferably formic acid, at a pH in the range of 2–4 for about 16–24 hours at 10°–30° C. Following the swelling, the material is mechanically subdivided in various types of apparatus ranging from a meat grinder to a colloid mill. Final solution is obtained by adjusting the pH to 2–4 by the addition of organic acid, filtration being employed after these operations to remove any remaining fibers. Such a solution free from fibers or fibrous material may be extruded through small orifices and formed into very fine filaments of exceptional uniformity and purity to render them especially adaptable for use as sutures.

Said collagen solution is of 5–15% collagen content, preferably 9–10%, and is introduced under

pressure at inlet 1 of Figure 1 for extrusion through orifices 2 contained in spinnerette 3 held in spinnerette head 4 by threaded member 5 which is turned to effect a seal between the spinnerette and flanged feed line 6 using annular gaskets 7. The spinnerette head is heated by focusing infra-red lamp 8 thereon. The collagen filaments 10 formed by such extrusion are pulled through coagulating solution 11 contained in tank 12. Preferably the coagulating solution is a concentrated ammonium sulfate solution containing at least 35% of said salt; and said salt solution is maintained at a pH of about 7.8 and a temperature of 20°–30° C. Such 5–15% collagen solutions are adapted to be extruded through spinnerettes having orifices of from 50 microns to 1 mm., although when preparing suture material it is preferable that orifices of 75–250 microns in diameter be employed. Also, it is preferable if the spinnerette head and the spinnerette itself are blackened, as, for example, by depositing a layer of carbon black thereon, in order to increase the efficiency of heating by directing infra-red rays thereon. The newly formed collagen filaments are pulled around pulley 13 and thereafter wrapped a few times about godet 14 which is operated in combination with take-up spool 16 to impart the desired degree of stretch to such collagen filaments.

Any suitable coagulant may be employed, although ammonium sulfate is preferred because of its high solubility (about 42%) and low cost. Generally, this coagulant is employed at a concentration of at least 35%. Sodium sulfate and magnesium sulfate can likewise be employed but being considerably less soluble are less effective. Various organic compounds have been suggested, such as ethyl alcohol, acetone, ethylene glycol mono ethyl ether, diacetone alcohol and the like. However, such expensive organic compounds possess no appreciable advantage over the less expensive inorganic salts.

The coagulated collagen filaments are thereafter spooled and subjected to suitable processing steps such as stretching, setting the stretch by contacting with coagulating solution for an extended period of time, further stretching, hardening and final drying. Thereafter such filaments may be twisted to form multi-filament twisted threads which may be used as such, or if desired may be used in the preparation of twisted plied strands or braided strands.

In Figure 2 which shows an arrangement adapted to heating by employing suitable fluid media, collagen solution is introduced at inlet 1 and thereafter forced under pressure through orifices 2 contained in spinnerette 3. As before, the spinnerette disc is held in place in spinnerette head 4 by bezel 5 which is threaded to permit its being turned to force the spinnerette against the annular gasket 7. Jacket 21 provides conical space 22 through which the aforementioned fluid media may be passed to heat the spinnerette head by introducing said media at inlet 23 and withdrawing the same at outlet 24.

In Figure 3 where an arrangement suitable for heating by electrical means is shown, collagen solution is similarly introduced at the small end of the cone, that is, at inlet 1 and likewise forced under pressure through orifices 2 contained in spinnerette 3 which is held in place by bezel 5 against annular gasket 7. The walls 31 of the spinnerette head 4 are wrapped with electrical heating wires 32, electrically insulated from said walls by a layer of mica 33, the whole being covered by asbestos sheeting 35, on top of which is wrapped a layer of water-proof covering 36, and held in place by clamps 37 and 38. The lead wires for supplying the electrical current are shown at 40 and 41.

What is claimed is:

1. A process of producing a collagen filament which comprises extruding a solution containing substantially 5–15% collagen through a spinnerette head maintained above substantially 35° C. and into a spinning bath maintained at substantially 20° C. and removing the filaments therefrom.

2. The process of claim 1 in which the spinnerette is heated by means of infra-red rays.

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