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PRODUCTION OF COLLAGEN STRANDS

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This invention relates to new and useful improvements in the production of collagen strands, and is a continuation in part of our co-pending application, Serial No. 152,014, filed March 25, 1950, now abandoned.

It has been proposed to produce strands from collagenous material such as tendons. These strands, however, did not have the strength of quality required for many purposes. In accordance with one proposed process, the tendons or pieces thereof were directly subjected to the action of an acid to loosen the coherence of the individual fibers of the bundles and thereafter pressed out into bands. The acidulation in this manner, however, seriously weakened the fiber structure and thus the strength of the end product produced.

One object of this invention is the production of collagen strands which are characterized by their enormous strength and ability to withstand stresses and which may be used as the substitute for high-grade catgut. This, and still further objects, will become apparent from the following description.

The term "strands" is used herein to generically designate any elongated members such as filaments, strings, threads, etc.

The starting collagenous material for the formation of the strands in accordance with the invention comprises animal tendons, such as cattle or horse tendons.

In accordance with the invention the starting animal tendons are disintegrated into fibers, preferably while adding sufficient cooling water to prevent heating and to thereby produce a very thin fiber suspension. This fiber suspension, after the separation of any surplus water, is acidulated by mixing with acid to a pH of between 2.5 and 4.5 to form a swollen mass. The swollen mass is then diluted with water to form a castable mass which is cast into a thin foil. The foil is then formed into a strand as, for example, a filament or thread by splitting the same into narrow strips and twisting the strips.

The starting tendons are preferably freed from the flesh residues and, if necessary, from the sheaths and are cut into small pieces. These pieces are then disintegrated into the fibers as, for example, with the use of mills or other suitable arrangement as, for example, toothed-disc mills, wing beater mills, hammer mills, pan grinders, edge mills, or rolling mills with rifled rolls. If the tendons are salted, they should be first washed free from the salt prior to the disintegration.

The disintegration proper as, for example, in the mill must be effected so that substantial heating of the material does not occur. The disintegration is preferably effected with simultaneous cooling as, for example, by feeding a sufficient amount of cooling water into the mill to avoid heating of the fibrous mass produced and to form a thin fiber suspension.

The initial disintegration must be effected by a purely mechanical process and chemicals such as acids should not be used.

The fibrous material from the disintegration as, for example, the thin fibrous suspension from which the surplus water has been removed as, for example, with the use of a strainer, centrifuge or filter press is subjected to acidulation.

The acidulation may, for example, be effected with hydrochloric acid and, if necessary, with the addition of water while the fiber pump is being worked up as, for example, in a kneader-mixer or vessel provided with an agitating mechanism or the like.

The acidulation must be effected at a pH between 2.5 and 4.5, and preferably at a pH of about 3.5.

By effecting the disintegration of the tendons into fibers mechanically, as described above and without the use of chemicals such as acids, and by only effecting the acidulation after this disintegration using a pH between 2.5 and 4.5, the fibrous material is converted into a uniformly swelled mass which has the appearance of a homogenous turbid or translucent mass, the viscosity of which may range according to dry content from that of a thin machine oil to that of a dough which is just capable of flowing. The swollen mass may, for example, somewhat resemble a white gell or slime.

By effecting the acidulation in the manner described, the water present in the fiber pulp is absorbed by the fibers, causing them to swell and forming a uniformly swollen mass from which the swelling water can no longer be mechanically removed as, for example, by being filtered off or pressed off.

If the swollen mass formed by acidulation has not been made sufficiently homogenous by the working up in the beater or mixer, it may be further worked and homogenized by treatment as, for example, on a pair of rollers or by pressing through nozzles or perforated plates.

The swollen mass is then diluted with water to form a castable mass which should have a dry matter content of between 0.5 and 2%, and preferably between 0.5 and 1%. The addition of this water to form a castable mass is preferably effected with simultaneous intimate mixing as, for example, in an agitating mechanism. The castable mass thus formed appears homogenous and the individual fibers cannot be observed. The mass, for example, appears as a white, translucent liquid having an oil-line consistency.

If desired, materials which influence the characteristics of the strands as, for example, disinfectants, plasticizers, oil or fat emulsions, tanning agents, etc. may be introduced into the castable mass. The introduction may be effected simultaneously with the introduction of the diluting water as, for example, by evenly distributing the material in the water. Conversely, the material may be added to the castable mass after the same is formed or separately at the same time the water is added.

The castable mass should be freed from any air present as, for example, by allowing the same to stand, by centrifuging or the like. In the case of castable masses having a higher solid content as, for example, 1–2%, the removal of the air is somewhat more difficult and high speed centrifuging may be required.

The casting of the mass into the thin foil may be effected in any conventional manner as, for example, by spreading the mass in the form of a thin film on a support as, for example, a casting band and drying it. The casting is, however, preferably effected in a conventional film casting machine as, for example, by introducing the mass into the casting frame.

The drying of the film may be effected in any conventional manner as, for example, by blowing heated air over the mass, passing the casting band through drying channels or the like. The thickness of the film or foil to be produced may be adjusted by adjusting the dry content of the castable mass, adjusting the casting slot, etc. Thicker films may be produced by spreading the castable

mass in two or more layers on the dried films. The films of, for example, the thickness of about 10μ have been found preferable in the further working up into catgut and similar products.

After the casting, the foil produced may, for example, resemble a piece of cellophane if the casting is effected on a smooth support such as a glass surface. When the casting is effected on a casting machine, the thin foil ribbon has a somewhat textured appearance.

The foil may be wound up on spools for storage or may be immediately further worked up.

For the purpose of producing strands such as catgut, strings for musical instruments, tennis racquets, textile filaments for special purposes and the like, the foil coming from the casting band in the dried condition or unwound from a storage spool may, for example, be cut into narrow strips, for example 1–30 mm. in width in a longitudinal direction. The cutting may be effected, for example, with a conventional film cutting machine. The width of the narrow strips depends on the desired thickness of the end strands to be produced. Narrow strips will, of course, form thinner strands and wider strips, thicker strands. Strands are then produced from the narrow strips in a known manner by twisting, i. e. spinning the strips, which have preferably been moistened.

The moistening of the strips may be effected with pure water or with water to which suitable additives have been incorporated as, for example, alcohols for inhibiting swelling, basically active substances counteracting the acid swelling, disinfectants such as iodine or iodine compounds, reducing agents, tanning agents and the like. These additives may be employed separately or in any desired combination.

The spinning may also be effected by twisting a number of strips together to form a single strand. It is thus possible to produce filaments of different thicknesses from narrow strips.

In order to produce strands of great strength, it is preferable to carry out the drying with increasing tension. The elemental fibers are thus directed more or less substantially parallel to the axis of the filament, i. e. linearly aligned, which produces a considerable increase in the breaking strength.

Upon drying, strands of great strength are obtained which may be employed, for example, as a substitute for catgut, as strings for musical instruments, tennis racquets and the like, or for special purposes in the textile industry.

The strands as, for example, filaments resemble high quality catgut and have a tearing strength in excess of 50 kilograms per square millimeter.

A particular important field of application for use of the strands produced in accordance with the invention is as surgical sutures which are reabsorbed in the human or animal body in exactly the same way as cutgut produced from sheep intestines. The rate of reabsorption can be adjusted by varying the degree of tanning. Untanned strands are reabsorbed much more quickly than catgut produced from sheep intestines. Such strands are consequently suitable for ligatures in tissues in which it is desirable that they disintegrate and be reabsorbed within a short time as, for example, within a few hours. As has already been stated, tanning or hardening may most suitably take place in accordance with the invention by adding tanning agents during the manufacturing process, particularly at the time of the formation of the castable mass. The finished, untanned strands may also be subsequently tanned with any known tanning agent, in which connection there are preferably employed tanning agents which do not have an immediate tanning agent but which can later be given a tanning action, as, for example, by a pH adjustment or by reduction as, for example, in the case of chromates such as sodium dichromate. The surgical strands produced in accordance with the invention are not subject to the sterilization difficulties of the conventional sheep intestine catgut.

The process in accordance with the invention is extremely simple and reliable and no disturbing decomposition of the collagen takes place.

The following examples are given by way of illustration and not limitation:

Example 1

Cattle and horse tendons were washed free from salt, freed from the flesh residues and sheaths and cut into small pieces. These pieces were disintegrated in a mill while adding sufficient cooling water to prevent heating. A thin suspension of the tendon fibers was recovered from the mill. This fiber suspension was freed from excess water with the use of a filter press, forming a fiber pulp. The fiber pulp was placed in a vessel provided with a mixer and hydrochloric acid and water were added to form a pH of about 3.5. After the addition of the acid the fibers commenced to swell, forming a uniformly swollen mass which had the appearance of a white gell or slime.

The swollen mass was intimately mixed with water in a vessel provided with an agitating mechanism. An amount of water was added to form a dry content of about 0.5–1%. Upon this dilution a castable mass which appeared as a homogeneous, white, translucent liquid having an oil-like consistency appeared. The individual fibers could not be observed in this castable mass.

The mass was fed into the casting frame of a conventional film casting machine and, after drying, a foil having a thickness of about 10μ was formed. The dry foil was cut into strips of about 15 mm. width. The strips were moistened with water and spun by twisting into a strand which had the appearance and characteristics of a high quality catgut.

Example 2

A swollen mass was produced in the manner described in Example 1 by the disintegration and acidulation of the animal tendons. The swollen mass was diluted in a mixer with water to a dry content of about 1.5%. The diluting water additionally contained polyethylene glycol amounting to 5% calculated on the dry collagen and having a molecular weight of 2,000. The castable mass thus formed was freed from air and cast on the band of a film casting machine. After drying, a foil of 25μ thickness was formed. The foil was cut into bands of 25 mm. thickness and five such bands were moistened and spun together by twisting into a thread. The added polyethylene glycol had an excellent softening effect on the strand which constituted a higher quality string suitable for the production of tennis racquets.

Example 3

A swollen mass was formed by disintegration and acidulation of tendons in the manner described in Example 1. The mass was diluted with water in a mixer to a dry content of 2%. The mass had a high viscosity and was freed from the entrapped air by cetrifuging at high speed. The mass was applied evenly under high pressure from a flat nozzle to the band of a flat casting machine. After drying, a foil was produced having a thickness of 40μ. The foil was cut into longitudinal strips having widths of 30 mm. and eight such strips were passed through a bath containing 10 parts of glycerin and 90 parts of water. The wetted strips were then spun by twisting into a thread and dried. The end thread had a thickness of 3 mm. and was polished on a polishing machine to a uniform thickness of 2.8 mm. The string produced constituted a higher quality strength suitable, for example, as a cello string.

The above description has been given in order to illustrate and not limit the invention, and various embodiments and modifications will become apparent to the skilled artisan which fall within the spirit of the invention and the scope of the appended claims.

We claim:

1. Process for the production of collagen strands from animal tendons which comprises mechanically disintegrating tendons into fibers, acidulating the fibers within a pH range of about 2.5–4.5 by mixing the fibers with acid to form a swollen mass, diluting said swollen mass with water to form a castable mass, casting said mass into a thin foil, and forming strands from said foil.

2. Process according to claim 1, in which said disintegration is effected while adding sufficient cooling water to prevent heating and thereby produce a thin fiber suspension, and in which the surplus water is separated from said fiber suspension prior to said acidulation.

3. Process according to claim 1, in which said swollen mass is diluted with water to form a castable mass having a dry matter content of 0.5–2%.

4. Process according to claim 1, in which said strands are formed by splitting siad foil into narrow strips, moistening the strips and forming filaments from said strips by twisting.

5. Process according to claim 1, in which said acidulation is effected to a pH of about 3.5.

6. Process according to claim 1, in which said swollen mass is diluted with water to form a castable mass having a dry matter content of 0.5–1%.

7. Process according to claim 1, in which a tanning salt of chromium is incorporated into the strand.

8. Process according to claim 1, in which a tanning agent is incorporated in latent form into the strands during the formation thereof and is later rendered active in the completed strands.

9. A process for the production of filaments and strings from animal tendons which comprises disintegrating tendons into fibers while adding sufficient cooling water to prevent heating and thereby produce a very thin fiber suspension, separating and removing the surplus water from said fiber suspension, mixing the fibers with acid to a pH range of 2.5–4.5 to form a swollen mass, diluting said swollen mass with water to form a castable mass having a dry matter content of 0.5–1%, casting said mass into a thin foil, slitting said foil into narrow strips, and forming filaments from said strips by twisting the strips.

10. Process according to claim 9, in which a tanning salt of chromium is incorporated in the material.

11. Process according to claim 9, in which a tanning agent is incorporated in latent form into the filaments during the formation thereof, and is later rendered active in the completed filaments.

No references cited.