Sept. 19, 1944.   D. TRAILL   2,358,427

MANUFACTURE OF FILAMENTS FROM VEGETABLE GLOBULIN

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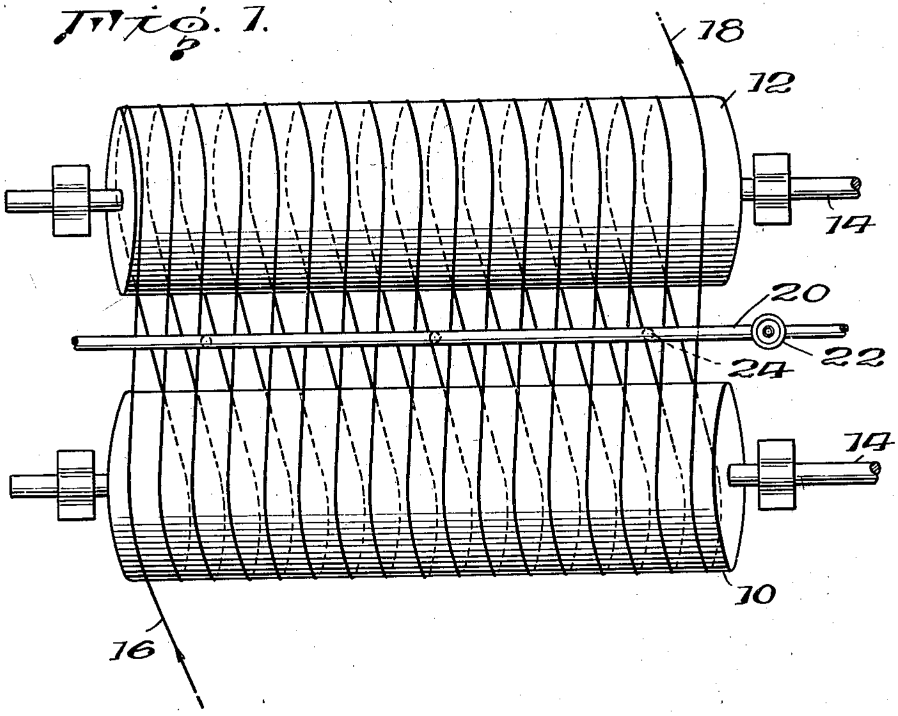
Fig. 1.



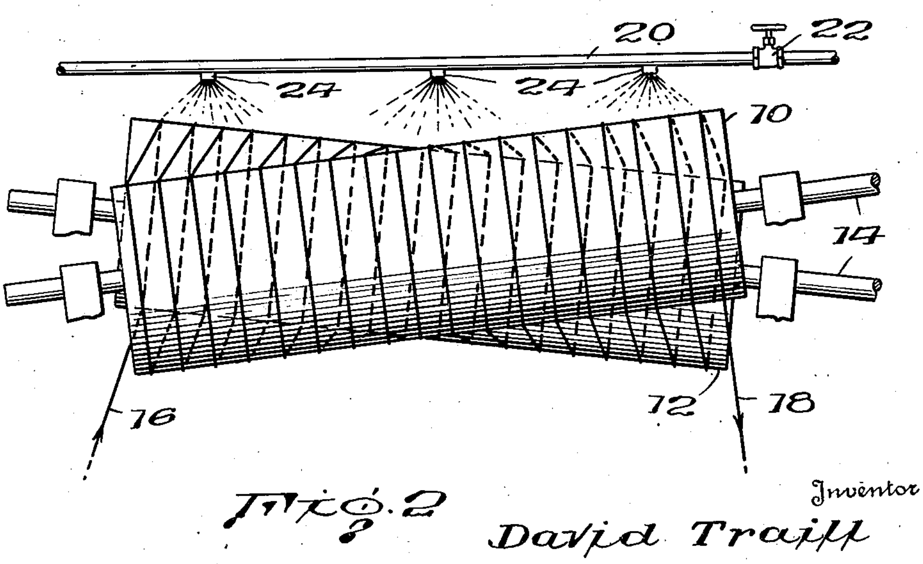


Fig. 2

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MANUFACTURE OF FILAMENTS FROM VEGETABLE GLOBULIN

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4 Claims. (Cl. 18—54)

The present invention relates to the manufacture of filaments by the extrusion of a solution of a peanut protein in dilute aqueous alkali from a spinnerette or the like into a coagulating bath. Suitably prepared solutions of peanut protein in dilute aqueous alkalis can be coagulated in the form of continuous filaments by extrusion into coagulating baths comprising an aqueous solution of an alkaline metal sulphate or other metal sulphate or ammonium sulphate, acidified with sulphuric acid, which may also include other substances; but in order to obtain strong filaments it is necessary that the coagulation should take place while the thread is wound out of the bath at a faster rate than the solution is extruded. The speed at which the filaments are wound may conveniently be about 3 to 8 times that at which the alkaline solution is extruded.

Filaments so obtained, however, are not yet chemically suitable for textile employment since whenever they are washed with water sufficiently to effect a suitable reduction in the salt concentration they commence to swell excessively and cannot be dried satisfactorily. In order to reduce their capacity for swelling in water and render it possible to wash and dry them it is therefore necessary to treat the filaments with a hardening agent, which is usually formaldehyde. Formaldehyde treatment alone still leaves the filaments somewhat lacking in resistance to wet processing, and in order to render them more water repellent and resistant to the action of boiling acid baths such as are used in wool dyeing, it is customary to subject the filaments to treatment with other reagents. Such treatment may be carried out in a mixed bath in conjunction with the formaldehyde treatment or in the form of some subsequent treatment.

While it has been suggested to include formaldehyde in the coagulating baths used in the manufacture of protein filaments, the action of formaldehyde in hardening the coagulated peanut protein is much slower than the coagulation of the peanut protein into the form of filaments, and it is more convenient to carry out the hardening as a subsequent operation after the coagulated filaments have been wound up. The filaments are commonly washed with substantially saturated brine, in contact with which material they are kept until the hardening treatment is commenced.

In spite of the fact that the filaments are wound out of the coagulating bath at a faster rate than the solution is extruded through the spinnerette the tension at which they are wound is only small, this tension being due to the weight of the filaments themselves, the surface tension at the spinnerette, and the frictional resistance between the filaments and the liquid of the bath and between the filaments and the guides across which they pass. If this tension is relaxed the filaments contract strongly even if they are placed in a concentrated solution of common salt in which they can be stored without swelling.

It is possible to prevent this shrinkage by holding the filaments at the desired length by mechanical means during the hardening period and any previous storage. But this hardening period is a period of usually 20 hours and the mechanical difficulties involved on a large scale plant would be almost insuperable.

It has been suggested to harden casein fibres in a sequence of solutions containing various proportions of 12% sodium chloride and 40% formaldehyde, the proportion of formaldehyde being low in the early baths in the sequence and rising in each bath in succession. The filaments are kept under tension in the first bath to avoid shrinkage. Such a process would not be applicable to peanut protein which would be dissolved in salt concentrations of this order.

It has also been suggested to harden vegetable albumin filaments by conveying them, preferably under tension, through two baths the first a sodium chloride bath of unspecified strength, to which aluminum salts can be added, preferably at a temperature of between 35° C. and 50° C. and the other a bath containing aluminum salts and sodium chloride at a temperature between 55° C. and 65° C. The effect of this process on the subsequent shrinkage of the filament in the hardening bath is undisclosed while the process would not be applicable to peanut protein except in very special circumstances.

This invention has as an object to provide a method which will reduce the shrinkage of peanut protein fibres when they are kept in contact with saturated brine. A further object is to provide such a method which will be rapid, economical, and easy to operate without damage to the fibre. Further objects will appear hereinafter.

These objects are accomplished by the following invention.

We have found that if the peanut protein filaments are maintained by mechanical means at the length at which they were wound up from the coagulating bath, being treated meanwhile with substantially saturated brine, the contraction on removing the filaments from the mechanical means maintaining their length is greatly reduced and that the hardened filaments obtained thereafter are characterised by improved tensile strength and increased elongation at break.

According to my invention the filaments after being wound up out of the coagulating bath are maintained at substantially the same length by mechanical means while being treated with substantially saturated brine.

The treatment with brine can either be applied as a separate treatment or simultaneously with the storage or the hardening treatment.

The longer the treatment with brine while mechanically held, the more effective it will be to reduce the shrinkage, but the major part of the effect takes place in the early stages. For practical working we have found that a treatment of 5 mins. is sufficient but we prefer to treat for 10 mins. We have obtained the best results by using completely saturated brine and find that any dilution of the brine results in damage to the fibre. As the fibre when treated is still wet from the coagulating bath it is difficult to assess the exact strength of the brine which is in contact with the filaments to within 3% but we have found that if the strength of the brine in which the wet filaments are immersed is reduced from 26.5 gms. of sodium chloride per 100 gms. of solution (saturated solution) to 23 gms. per 100 gms. of solution there are noticeable signs of damage to the fibre. The filaments after hardening and drying have a harsher handle, are to a great extent stuck together and have to be teased out before they can be used for textile purposes, whereas the treatment with saturated salt leaves the bundles of filaments open after hardening and gives a soft handle. Treatment with 20% salt solution results in fibre badly stuck together. In one case after 10 minutes treatment with completely saturated brine the shrinkage, after the filaments had been released from the mechanical means holding them, had been reduced from about 50% to 19%. With 23% salt solution the reduction was only to 24% and with 20% solution to 29%. The improved strength, and elongation at break of the hardened filaments appears to be given by this process if 23% salt solution is used but 20% solution is not so effective.

If desired the filaments advancing from the coagulating bath may be introduced in a continuous manner into contact with the brine while they are still advancing instead of being wound up and subsequently treated while stretched on a tensioning frame. If desired apparatus of the known skew roller or intermeshing cage type adapted to advance the filaments in a generally helical path may be employed to assist the filaments to remain in contact with the brine in their extended condition for the required length of time. Such apparatus may be immersed or partly immersed in a brine bath or the brine may be distributed over the filaments extended on the apparatus.

One preferred form of apparatus is illustrated in the accompanying drawing wherein

Fig. 1 is a more or less diagrammatic elevational view and

Fig. 2 is a plan view of a pair of rollers arranged for utilization in accordance with the present invention.

It will be seen that rollers 10 and 12, more or less identical in construction, are mounted upon suitable bearings 14 and are adapted to be driven by any suitable means, not shown. It will also be seen that the rollers are disposed at a suitable relative angle and so arranged as to receive the oncoming fibres 16 at the left-hand-most extremity, as viewed in the figures and to advance the fibres progressively and in a more or less helical path toward the opposite extremity where they are discharged as at 18.

A spray device comprising a conduit 20 under the control of a valve 22 and having a plurality of spray heads 24, is adapted to distribute the salt solution over the rollers. Thus the fibres being advanced along the rollers are continuously contacted and drenched by the concentrated brine through their period of residence upon the rollers. As pointed out hereinabove, the rollers are driven at identical speeds in order to advance the fibre under a fixed degree of expansion and thus resist shrinkage or contraction. It will be furthermore evident, as stated above, that the present illustration is more or less diagrammatic and that in practice the proportions of the parts are so adjusted as to maintain the fibre for the desired length of time in contact with the solution, additional pairs of rollers being utilized in serial relationship if necessary.

The invention is further illustrated by the following example:

Example

A solution of peanut protein in dilute aqueous caustic soda is extruded through multiple spinnerettes at such a rate into a bath containing saturated sodium sulphate acidified with sulphuric acid that when collected at a rate of 75 metres per minute a linear extension of five-fold occurs between the spinnerette and the collecting device. The bundles of filaments run from the coagulating bath over a guide directly on to the first of a series of pairs of cylindrical rollers rotating in the same direction on skew axes so that the filaments pass round each pair of rollers several times in a generally helical path and on reaching the end are led to the next pair of rollers in the series. The surface of each pair of rollers is sprayed with saturated brine. The angle of skew and the number of rollers is such that it takes ten minutes for the filament advancing on to the first pair of rollers to reach the end of the last pair. This time is considerably longer than the time required to reduce the sodium sulphate concentration to a negligible amount. The rope of filaments is then cut into the desired staple length and stored in contact with saturated brine, after which it is centrifuged to remove surplus brine and introduced into about twenty times its weight of a solution of saturated brine containing 1% of its weight of formaldehyde and 1.3% of its weight of hydrogen chloride, in which it is maintained at 35° C. for 24 hours. The fibre is then washed with water until it is practically free from salt. The fibre may then be made faintly alkaline by treatment in a dilute sodium carbonate bath, washed and dried.

The filaments prepared according to the example have a soft handle and have good elongation. They are resistant to wet processing and can be dyed in acid dye baths such as are used for wool dyeing, and may be used in textile operations.

This invention is a valuable advance in the art as it reveals for the first time a method whereby a peanut protein filament can be made resistant to hot water and acid dye baths without undue shrinkage taking place in the resistance-giving process, without recourse to elaborate and costly machinery, without damage to the fibre, and with the use only of ordinary temperatures and a cheap and easily available material, namely common salt.

As many seemingly widely different embodiments of the invention will be apparent without departing from the spirit and scope thereof it must be understood that the invention is not limited to any specific embodiment except as defined in the appended claims.

I claim:

1. In a method of manufacturing peanut protein filaments which comprises extruding a liquid containing a solution of peanut protein in dilute aqueous alkali into a coagulating bath whereby the extruded liquid is coagulated to form a filament, drawing the coagulated filament from the bath at a rate between 3 and 8 times the rate of extrusion to stretch said filament and subsequently treating the filament with a hardening agent adapted to render it resistant to hot water and acid dye-baths, the steps which comprise treating the coagulated and stretched but unhardened filament with a substantially saturated aqueous solution of common salt while maintaining by mechanical means the stretch imparted thereto as it is drawn from the bath, whereby to provide a filament having a reduced tendency to shrinkage, releasing said filament from said mechanical means and thereafter treating said filament with said hardening agent.

2. The step claimed in claim 1 in which the substantially saturated solution of common salt contains at least 23% of common salt.

3. The step claimed in claim 1 in which the maintenance of the length by mechanical means and the simultaneous treatment with substantially saturated salt solution is carried out for at least five minutes.

4. In a method according to claim 1 wherein the hardening treatment is carried out while the filament is unsupported against mechanical shrinkage.

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