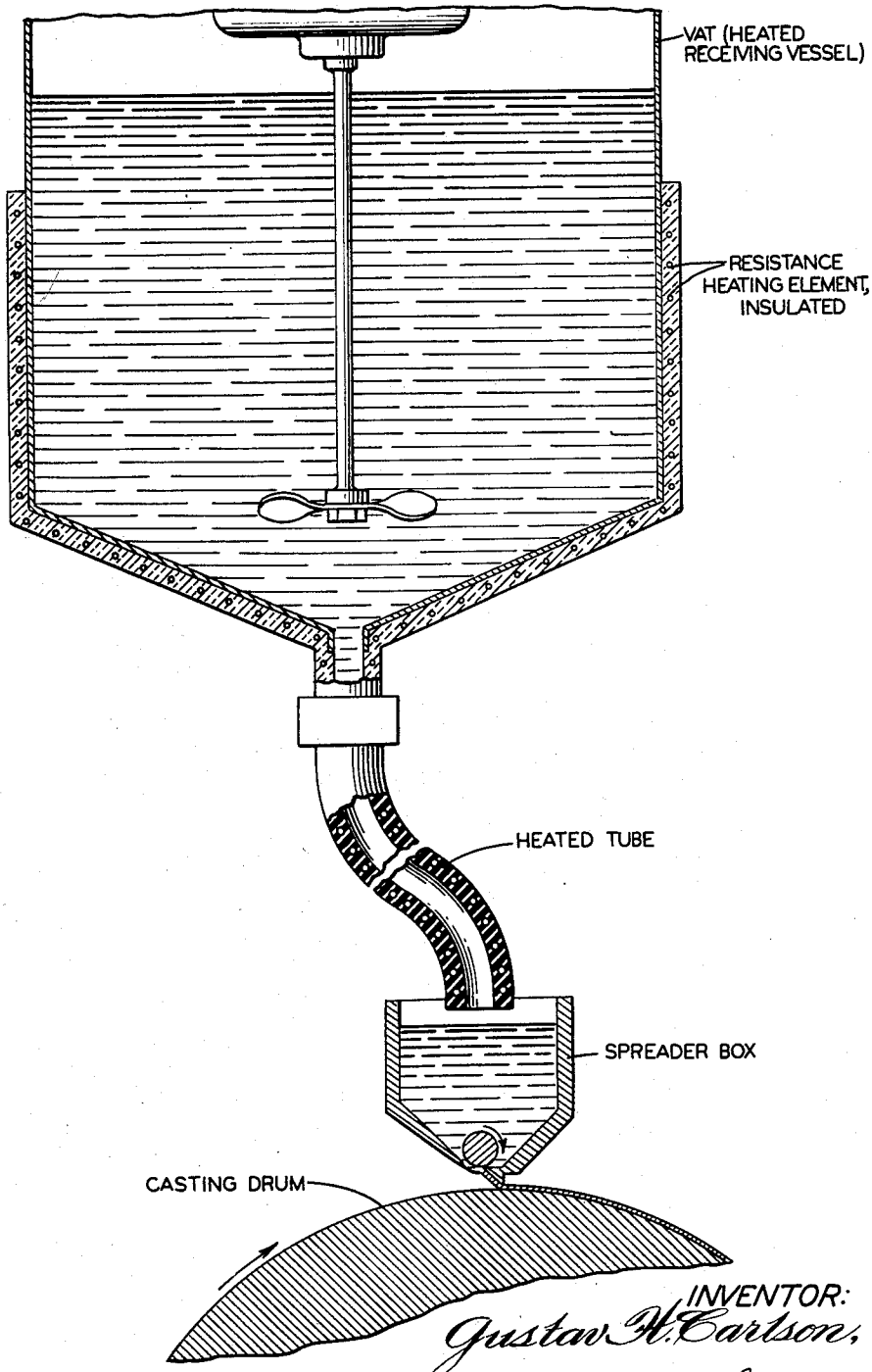
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PROCESS OF IMPROVING ADHESIVITY OF GELATIN USED IN PREPARING CAPSULES BY CONTINUOUSLY AGITATING THE GELATIN UNTIL CASTING

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PROCESS OF IMPROVING ADHESIVITY OF GELATIN USED IN PREPARING CAPSULES BY CONTINUOUSLY AGITATING THE GELATIN UNTIL CASTING

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This invention relates to an improved method for preparing soft gelatin capsules from a fresh gelatin mixture or a gelatin composition containing reworked gelatin net. The method of the invention is particularly adapted to the formation of capsules filled with a liquid or semi-liquid content on a continuous basis. Apparatus suitable for continuous production of capsules has been disclosed previously in the literature and comprises a pair of smooth faced drums on each of which a gelatin ribbon or film is cast, a pair of cooperating die rolls containing complementary die cavities in which the ribbon is shaped to form the capsules, and a feeder head disposed in the bight of the die rolls for introducing the liquid content into the capsule as it is formed. The ribbons are stripped from the drums and simultaneously fed over the rotating rolls into juxtaposition whereupon the liquid content is ejected, at precisely the correct time, from the feeder head into the ribbons to stretch the same into opposed die cavities to shape and fill the capsule. As the die rolls rotate, ridges surrounding each of the cooperating opposed cavities serve progressively to seal the ribbon together about the entire periphery of the distended portion of the ribbon which comprises the capsule. It is evident that the success of this process depends in large part upon the ability of the gelatin webs or ribbons to adhere together to form a seal. Failure of this seal at any point about the capsule periphery will result in a leaky capsule.

What remains of the gelatin ribbon or web after the material required for the capsules has been severed therefrom is known as "net," since it resembles a net in appearance. It is customary to rework the net either by direct remelting and reuse or by incorporation with gelatin masses prepared directly from fresh granular or flake gelatin containing plasticizer, flavors, dyes and preservatives. The gelatin compound is then melted and placed in a large heated vat from which it is discharged into smaller hoppers or spreader boxes mounted above the drums on which the gelatin is cast to form the ribbons. In commercial production of capsules, this vat will normally hold sufficient gelatin to supply the capsulating machine for 16–24 hours, depending upon the thickness of the ribbon film. The vat is heated to maintain the gelatin at a temperature of 125–140° F., preferably 135° F., which keeps it at the proper casting viscosity. It has been found that under these conditions, the last one-quarter or one-third of the gelatin in the vat will produce sheets that do not seal satisfactorily. To avoid leaky capsules, this material hitherto has been discarded. Although some of it may be reworked, this procedure is expensive and wasteful due to the labor involved in handling the material, the increased size of the vats necessary to supply a day's run, and the possibility of decreased quality in the reworked batch.

The reason for lack of adhesion in the last portion of gelatin to come from the vat is not known for certain and we do not want to be held to any theoretical explanation since such explanation is not absolutely necessary for

an understanding of the invention. However, it is believed that depolymerization of the gelatin or some degrading action akin thereto results from heating the gelatin in the presence of a catalyst introduced into the gelatin mix with the net. The last portion of gelatin dispensed from the vat is characterized by reduced viscosity, low Bloom strength (may decrease to one-half or less of the original value, or the material may even liquefy), and occasionally by separation of the water and plasticizer. All of these properties indicate a break down of the protein molecule. Furthermore, the degraded material has a low specific gravity and rises to the top of the vat so that this is the portion that is last to be used. It appears that degradation of the protein may well be accelerated by traces of catalyzing metals, such as copper, nickel, cobalt, iron, molybdenum and the like, which are present in the liquid content of many capsules, particularly vitamin capsules containing trace minerals. As the capsules are formed minute traces of the liquid capsule content material may contaminate the net. This slight contamination is unavoidable in as much as the interior surface of the net wipes past the injection orifices of the injection element or wedge. Only minute traces of such metals are necessary to catalyze degradation and such traces will remain in the net in spite of washing and other precautions that might be taken. Furthermore, dyes incorporated into the gelatin to color it may likewise catalyze degradation of the gel.

Regardless of what causes degradation and separation of the constituents of gelatin compounds, especially those containing redissolved net, we have discovered that the difficulty can be obviated by thoroughly stirring the vat of gelatin so that the quantity being dispensed from the vat is completely and constantly agitated until the casting operation. The agitation apparently prevents orientations and polymerizations of the protein molecules and thus terminal amino and carboxyl groups, and possibly more so, secondary molecular attractions, remain activated to produce tenacious seals, for gelatin compositions which have been thoroughly agitated remain tacky and seal tightly even after 18–24 hours at elevated temperatures in the range of 125° to 145° F. Thus, large vats of gelatin may be used to the last drop. The savings effected are at once obvious.

The single figure of the drawing illustrates an apparatus suitable for carrying out the invention. The gelatin may be mixed by an electrically operated stirrer disposed within a vat, the bottom portion of which is heated with a resistance heating element or other suitable source of heat. The thoroughly agitated gelatin composition is conducted from the vat to the spreader box through a heated tube. As shown in the drawing, the tube may be heated by means of an electrical resistance element. The spreader box is mounted just above the casting drum and serves to deposit a thin film of gelatin on the surface of the drum. A rotating spindle within the spreader box insures uniform distribution of the gelatin from the slot in the bottom of the box.

To illustrate the invention, the following specific example is given. A gelatin compound for producing a gelatin shell of medium hardness may be prepared by mixing 43.85 parts of gelatin, which includes about 15 parts of naphtha-washed gelatin net, the rest being fresh gelatin flakes, with 20.80 parts of glycerin and 20.93 parts of water (all parts being by weight). The weights given are not absolute but include water normally held by the glycerin and gelatin. A small quantity of preservative was also added. The materials were blended in a pony mixer until the gelatin had picked up the liquids and was thoroughly soaked. This required from 10–30 minutes. The mixed mass containing the swelled gelatin globules was then poured into a gelatin melter, such as

that shown and described in Patent No. 2,533,964 to J. O. Scherer. This melter consists of a steam-jacketed vessel having a convex heated bottom mounted on a smaller heated receiving vessel. The receiving vessel normally will hold about 30–40 gallons. A narrow annular space between the convex bottom and the side walls of the melting vessel permits the molten gelatin to flow therethrough into the heated receiving vessel as it reaches melting temperature. The melter is connected to a source of vacuum in order to remove all of the air from the gelatin mass. The temperature of the jacket surrounding the receiving vessel and the melting vessel is sufficiently high to raise the temperature of the gelatin to from 130–160° F. Melting approximately 30 gallons of gelatin requires about 4–6 hours. After the gelatin mixture was melted in this manner, suitable dye was added and thoroughly mixed into the gelatin mixture. The heated receiving vessel serves as the vat which supplies the gelatin to the capsulating apparatus. For details relative to the construction of a suitable capsule forming apparatus see Patent No. 2,288,327 to R. P. Scherer. In accordance with the present invention the melted gelatin mass was constantly agitated in the vat while maintained at approximately 135° F. The gelatin flowed from the vat through a heated tube into the hoppers or spreader boxes mounted on the casting drums on which the ribbons are cast. The gelatin dispensed from the vat after 16–18 hours at 135° F. was found to have excellent properties. The capsules prepared from the cast ribbons were of a quality equal to those prepared from gelatin first to flow from the vat.

A second batch of gelatin was prepared as in the example just described, except no gelatin net was employed. The 43.85 parts of gelatin consisted entirely of fresh gelatin flakes and the proportions of the other ingredients were correspondingly adjusted as follows: glycerin, 24.47 parts and water, 20.93 parts by weight. The procedure described for melting, casting and forming capsules was repeated using this composition with the same results. There was no noticeable deterioration in the quality of the capsules prepared from the last 25% of gelatin remaining in the vat after 16–18 hours at 135° F. The adhesion of the opposed halves was excellent.

What we claim as new and desire to secure by Letters Patent of the United States is:

1. A method for preparing gelatin capsules from a fluid molten gelatin compound characterized by loss of adhesivity upon aging at a temperature above its melting point which comprises thoroughly agitating said gelatin compound continuously until casting, casting a pair of gelatin ribbons from the mixture, then bringing the ribbons together to form leaktight capsules by adhesion of said ribbons.

2. A continuous method for preparing gelatin capsules from a fluid molten gelatin compound characterized by loss of adhesivity upon aging at a temperature above its melting point which comprises constantly agitating the gelatin compound to insure thorough mixing thereof, flowing the freshly agitated gelatin onto a pair of casting drums to form a pair of gelatin ribbons, and bringing the ribbons together to form leaktight capsules by adhesion of said ribbons.

3. The method of claim 1 wherein said gelatin compound contains redissolved gelatin net.

4. The method of claim 2 wherein said gelatin compound contains redissolved gelatin net.

5. In a method for preparing gelatin capsules by casting ribbons from fluid molten gelatin containing redissolved gelatin net and bringing said ribbons together to form capsules by adhesion thereof, said gelatin being characterized by loss of adhesivity upon aging at a temperature above its melting point, the step which comprises thoroughly agitating the molten gelatin just prior to casting the ribbon therefrom, thereby insuring adhesivity of the ribbon thus formed.

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