Nov. 7, 1961 D. T. MILNE 3,007,832

SEALING OF ALKALI SENSITIVE FILMS AND PELLICLES

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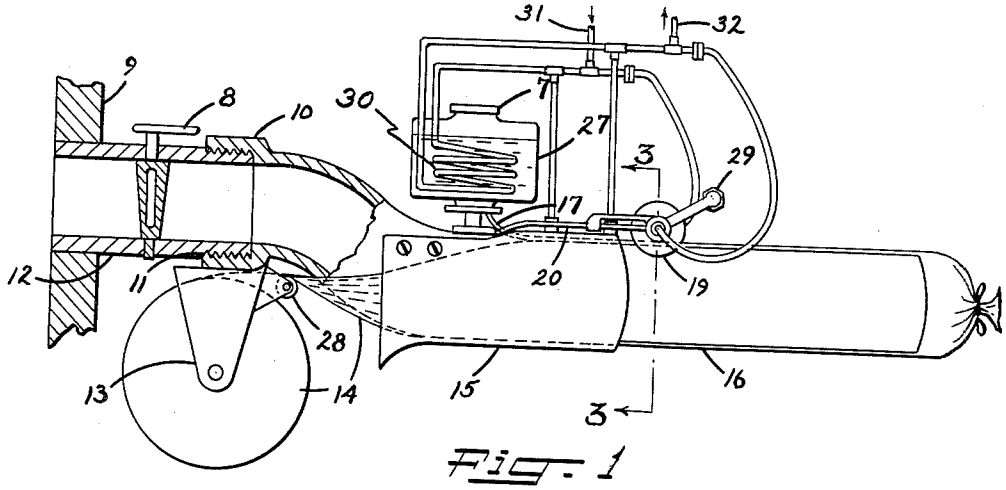


Fig. 1

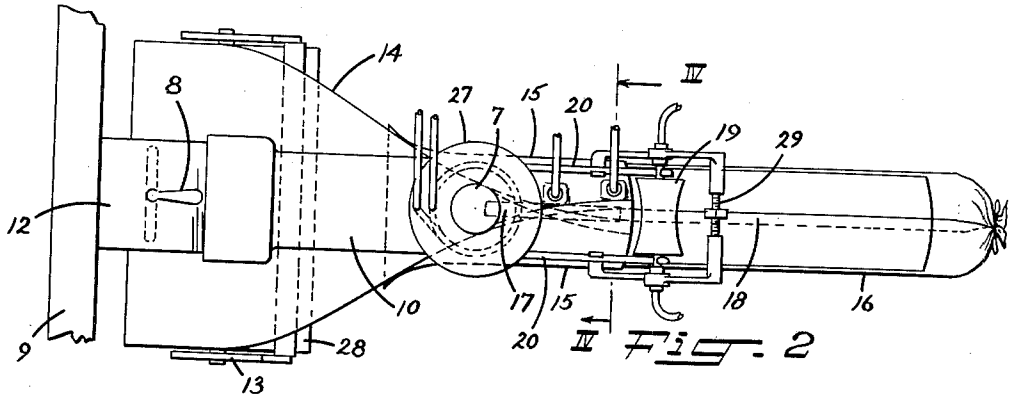


Fig. 2

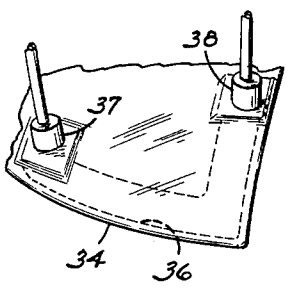


Fig. 5

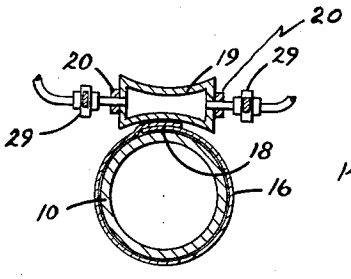


Fig. 3

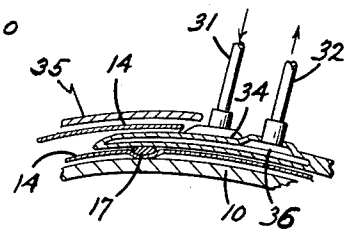


Fig. 4

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SEALING OF ALKALI SENSITIVE FILMS AND PELLICLES

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The present invention relates to the forming of seals for joints in films or sheets of alkali-soluble water-insoluble material, e.g., certain cellulose ethers which may be rendered tacky by the application of an alkaline solution to the surfaces of sheets or films thereof which are to be joined.

When films or pellicles, casings, bands, or other articles comprising a thin material containing an alkali-soluble ingredient are joined by the uniting of alkali-treated surfaces, it is generally desirable, because of the strength of the alkali solution used, to neutralize the alkali after such sealing so as to strengthen the joint, eliminate tackiness, and prevent contamination of other goods that may be brought into contact with other areas of the joints. The presence of strong alkali, if not neutralized, is particularly objectionable in the wrapping of meat. Furthermore, if, for example, casings having joints containing appreciable alkali are stored in a flattened condition, the alkali will leach into adjacent casings brought into contact with the joined portions to cause cohesion. Neutralization of the alkali may be accomplished by acid materials, such as dilute acetic acid or vinegar, but unless the casings are to be used immediately, any acid must be flushed from the casings or eventually it will embrittle and weaken the alkali-soluble material.

It is a primary object of the present invention to provide a process for sealing alkali-soluble sheet materials which may be accomplished with such a low concentration of alkali as to render neutralization unnecessary under the usual conditions of use of such sheets or films. Another object is to provide a process in which a high degree of adhesion is obtained practically instantaneously as the portions of the material forming the joint are brought together. Still another object is to provide a process in which a simple dilute alkaline solution which does not contain any other ingredient may be used. Other objects, features and advantages will become apparent from the following description of the invention and the drawing relating thereto in which:

FIGURE 1 is a side elevation partly in section of a form of apparatus with which the invention may be practiced;

FIGURE 2 is a plan view of the form of apparatus shown in FIGURE 1;

FIGURE 3 is a sectional view of the form of apparatus shown in FIGURE 1, the section being taken along line 3, 3 in FIGURE 1;

FIGURE 4 is a fragmentary view in section, taken along line IV—IV of FIGURE 2, of adjacent portions of tube-forming equipment; and

FIGURE 5 is a fragmentary plan view of a hollow edge portion of a tube former shown in FIGURE 4.

In brief, the invention comprises providing an alkali-soluble sheet or film of cellulosic material impregnated with urea whereupon a joint may be obtained between any desired surfaces of the material by simply wetting such surfaces with a solution having an alkaline concentration of from approximately 1 to 6 percent, pressing the surfaces together and drying them. In optional practice of the invention, alkaline solutions of very low concentrations may be more efficiently used by chilling the materials which form the joint. The urea may also be used as a plasticizer for the sheet or film and substituted

wholly or partly therein for conventional plasticizers. In many instances the alkaline solution may be applied in restricted amounts to eliminate a drying operation following the sealing operation.

As the drawing suggests, the method of sealing taught herein is readily adapted to the manufacturing of stuffed meat products since the method is particularly useful in forming tubular casings in which edged portions of a sheet are continuously brought into overlapping relationship, moistened with the dilute alkaline solution and sealed together to form a tube. As the sealing is instantaneous, such a casing may be stuffed and packed with meat within the same apparatus.

However, unstuffed casings which are completely dry and are prepared in a flattened condition for shipment to a user, or for storage for an indefinite period of time, may be prepared by an apparatus such as disclosed in my application, Serial No. 460,571, filed October 6, 1954, now Patent No. 2,845,357. Portions of the apparatus disclosed in the said application are provided for applying a neutralizer and would normally not be used in forming casings in accordance with the present invention. However, if a neutralizer is used, it can be applied by the neutralizer-applicator portion of the apparatus in greatly reduced quantity or concentration as compared to that required for sealing a cellulosic material containing no urea.

The seamed or jointed articles of thin gauge materials produced in accordance with this invention comprise alkali-soluble water-insoluble cellulosic materials, such as a lower alkyl hydroxy-alkyl, or carboxyalkyl ether of cellulose; preferred specific materials being methyl, ethyl, hydroxyethyl, and carboxymethyl ethers of cellulose, mixed cellulose ethers in this class; and also sheets derived from cellulose ether-xanthates, cellulose xanthoethers, cellulose thiourethanes, and cellulose xantho-fatty acids. Such cellulosic materials may be used in combination with a flexible base such as a porous paper. A preferred material, for example, that may be used in practicing the present invention is a thin porous paper thoroughly impregnated with an alkali-soluble cellulosic material, such as hydroxyethyl cellulose. Such a material is rendered immediately tacky by alkaline solution and in a condition for making joints by an alkaline solution of greater than 6 percent alkali.

However, joints may be made in a wrapping material constituted of any one or more of these cellulosic materials through the use of tackifying solutions containing less than 6 percent of an alkali when such material contains a small percentage of urea. The tackifying solution or adhesive used in sealing edges or portions of urea-impregnated alkali-soluble material may be a solution of sodium or potassium hydroxide or other strong base of less than 6 percent concentration which may contain in addition, but not necessarily, one of the alkali-soluble cellulosic materials, preferably that material comprising the members to be joined. In a preferred manner of practicing the invention, the amount of tackifying solution applied to the members to be joined is carefully controlled so as not to cause an excess of the solution to be squeezed from the joint and thus make necessary the use of a neutralizing medium and/or a drying operation. The amount of alkaline solution applied, for example, may be satisfactorily controlled by feeding it through a wick or other distributor held in contact with the surfaces to be joined, and applying thereto merely enough solution to moisten such surfaces.

After the portions of the sheet or film have been joined as the result of adhesion developed by the tackifying solution, they are preferably subjected to pressure to assure good union of the joined portions. If the joints tend to become brittle after thorough drying, as for ex-

ample, in the production, storage, and distribution in commerce of empty casings, a small amount of a plasticizer such as one of the polyhydric alcohols may be incorporated in the tackifying solution to provide sufficient plasticization within the joint.

Although urea alone may be used as a plasticizer for the sheet materials treated in accordance with this invention, it is normally more economical to use urea in that amount which results in good joints in combination with that amount of plasticizer which, together with the urea, results in good softening of the film or sheet. Although urea may be used in an amount as low as 1 percent of the dry weight of the film to noticeably reduce the concentration of alkali required in the tackifying solution, for effective use of the invention, concentrations of at least 3 percent are desired, and urea may be incorporated in the film or sheet to an amount up to as much as 30 percent of its dry weight for plasticization and sealing purposes. To obtain urea content in this range, the plasticizing bath through which the sheet or film is passed will have a urea concentration in the approximate range of from 1.5 to 12 percent. In using that amount of urea which produces efficient and instantaneous formation of joints, the remaining plasticizing requirement of the material may be supplied through one of the conventional plasticizers for cellulosic films, such as polyhydric alcohols, e.g., glycerine, glycol, diethylene glycol, sorbitol, and butylene glycol, or an alkylolamine. A typical commercial packaging material will contain a total of 15 to 25 percent of a plasticizer (based on the dry weight of the film). Of this amount, urea may amount to 3 to 20 percent with the remainder consisting of another plasticizer to provide satisfactory sealing of joints as required by this invention.

The following examples illustrate joints made in accordance with the invention as well as joints made with materials which do not contain urea so that comparisons may be drawn which demonstrate the effectiveness of the invention as claimed herein.

EXAMPLES I TO V

Films were prepared from a solution comprising 7.5 percent alkali-soluble water-insoluble hydroxyethyl cellulose, 7.5 percent sodium hydroxide and 85 percent water by casting this solution on a polyethylene belt by means of a reverse roll coater. The hydroxyethyl cellulose was a low etherified type in which ethylene oxide units were attached to the cellulose in a ratio of about one to 4 or 6 glucose units, i.e., a substitution factor of about 0.2. The film was coagulated in a solution comprising 11 to 15 percent sulfuric acid and 24.5 to 26.3 percent sodium sulfate at 40° C. Following coagulation, the film was washed in water at 75° C. and plasticized at 75° C. in plasticizing baths, the ingredients of which are listed in Table A below. After plasticizing, the film was dried on heated rollers at 75° C. The composition of the plasticizing baths and of the film after drying are as follows:

Table A

Example	I	II	III	IV	V
Composition of Plasticizing Bath:					
Glycerine, percent	7.5	7.5	2.5	2.5	2.5
Urea, do	0	0	6.0	6.0	4.0
38% Sodium 2-ethyl hexy sulfate (surface active agent), percent	0	0.5	0	0.5	0.5
Composition of Film:					
Urea, percent	0	0	15.1	16.0	10.0
Glycerine, do	19.9	19.5	6.4	7.4	6.6
Moisture, do	9.3	9.0	8.1	8.4	8.8
Cellulosic Material, do	70.8	71.5	70.4	68.2	74.6

Table B

COMPARISON OF STRENGTHS IN JOINTS AT 24° C.

Example	I	II	III	IV	V
NaOH in Tackifying Solution:					
2%	Very Poor	Very Poor	Poor	Poor	Poor.
4%	Poor	Poor	Extra Good	Extra Good	Extra Good.
6%	Good	Good	Excellent	Excellent	Excellent.
8%	Excellent	Excellent	do	do	Do.

Explanation of Terms in Table B:
Excellent—Tears the film in other place than seal.
Extra Good—Tight seal, slight tendency to tear the film rather than the seal.
Good—Medium seal.
Poor—Slight seal requiring slight pull to part the joint.
Very Poor—Very little, if any, seal; the joint parts with practically no pull.

In the above examples commercially satisfactory seals were obtainable with films having a urea content of 10 percent or higher with tackifying solutions as low as 3 or 4 percent concentration of alkali. On the other hand, the film samples of I and II contained no urea and produced poor seals when the joints were made when using a tackifying solution of 4 percent alkali; commercially acceptable joints were obtained only when the alkali concentration was increased to above 6 percent.

The above examples, while illustrative of the relative effectiveness of seals made in an alkali-soluble cellulosic material containing some urea in the one case and no urea in the other case, do not embody conditions which are conducive to most economical use of alkali and urea. For example, the seals of these examples were made at room temperature. The concentration of the alkali solution used for joining urea-containing hydroxyethyl cellulose may be readily reduced to 1 percent if sufficiently low temperatures are employed, e.g., a temperature of 10° C. or lower. Equipment for obtaining "cold seals" is described hereinafter. The chilling may be accomplished by applying refrigeration to the alkali solution in its reservoir, by chilling the surfaces (wet or dry) just before the joining thereof, and/or by chilling the joint immediately after its formation before the water of the alkali solution has evaporated or has opportunity to be wicked in any substantial extent away from the surfaces wetted by solution.

The above examples were conducted with hydroxyethyl cellulose of film grade which is made with relatively low etherization in order to obtain low sensitivity to water. However, the sealing of such films may be implemented at very low alkali concentrations in practicing the present invention by increasing the extent of substitution, short of water solubility, of ethylene oxide units on the glucose units of the cellulose in the original preparation of the hydroxyethyl cellulose, e.g., to any value up to 0.4 ethylene oxide units substitution per glucose unit. Hydroxyethyl cellulose up to this degree of etherization retains water insolubility. Within this range of etherization, sealing solutions having no more than 1 percent alkali are practicable.

Referring to FIGURES 1, 2 and 3 of the drawing in which is shown a form of apparatus with which this invention may be practiced, reference character **10** indicates a nozzle having a threaded end **11** adapted to screw on the outlet pipe **12** of a conventional sausage meat container **9** to replace the nozzle conventionally used thereon. Since the meat in the container **9** is customarily placed under hydraulic pressure, the valve **8** is adapted and arranged to control the flow of the meat through the nozzle in the usual manner. On the underside of the nozzle **10**, there is mounted a bracket **13** adapted to support a roll of casing-forming, urea-containing hydrophilic sheet material **14**. On each side of the nozzle there is mounted a tube-former **15** which encircles the nozzle and is arranged to shape the sheet material into a tubing **16** about the nozzle. There is also mounted on the nozzle **10** a liquid reservoir **27**, for an alkaline solution, having a lid **7** and means such as a wick **17** for applying this solution to at least one of the overlapping surfaces of the hydrophilic sheet to form a longitudinal seam **18**. An arcuate roller or shoe **19** is supported on a forked spring **20** and is positioned to apply pressure to the seam **18**.

It is desirable to feed the sheet material in such a position that the center and the edges of the sheet are displaced an equal distance from the longitudinal axis of the nozzle. This is done, as shown in FIGURES 1 and 2, by forming the nozzle with a reverse curve at the end adjacent to the meat container, and there is provided a guide roller **28** which is so positioned that its upper surface lies in the same plane with the axis of the free end of the nozzle **10**. Thus, when the roll of sheet material is passed over this roller, it is roughly at the same level as the center of the nozzle and is suitably positioned for the formation of the tubing. It is to be understood that the nozzle may be reversed so that the roll **14** is positioned on the top side of the nozzle while the alkaline solution reservoir is on the underside, in which case the seam is formed on the underside of the nozzle. Instead of using a wick **17**, an equivalent means such as a roller or disk which dips into the alkaline solution reservoir and then contacts an edge of the sheet material may be employed, and such alternative means is particularly adapted when the nozzle is reversed.

FIGURE 3 is a cross-section of the nozzle shown in FIGURE 1 and taken along the line **3—3** thereof, in which is illustrated the nozzle **10**, and the sheet material **16** surrounding the nozzle and overlapping at the top to form the seam **18**. There is also shown a roller or shoe **19** that presses the overlapping edges of the seam **18** against the nozzle **10**. The roller **19**, as shown, is made hollow whereby it may be chilled by means, such as refrigerated brine circulated therethrough, to implement the sealing operation. A turn-buckle screw clamp **29** holds refrigerant line connectors in place over the ends of coaxial hollow shafts of the roller **19**.

In forming a "cold" seal, further chilling of the materials from which the seal is formed may be accomplished by providing a cooling coil **30** inside the alkaline solution reservoir having extremities connected with a refrigerated brine supply and return lines **31** and **32** respectively. Referring to FIGURES 1, 2, 4, and 5 of the drawing, the under hollow edge portion **34** of the tube former **15** (in overlapping relation with the outer edge portion **35**) has a chamber **36**. The edge portion **34** has passageways through its walls to the chamber as provided at **37** and **38** for passing a refrigerant into and out of the chamber.

In the operation of this invention, the sheet material **14** is passed over the guide roller **28** through the forming means **15** and shaped thereby into a continuous tubing **16** having overlapping edges to form a seam **18**. Between the overlapping edges there is applied an alkaline aqueous solution from the wick **17**, and the edges are then pressed together by means of the chilled roller or shoe **19** of

arcuate profile bearing on the upper surface of the nozzle. After the seamed tubing **16** passes beyond the end of the nozzle **10**, the tubing is tied and then may be filled with sausage meat. The flow of the sausage meat is controlled by means of the valve **8** and if desired, the stuffed casing is twisted at intervals to provide sausage links.

Various changes and modifications may be made in practicing the invention without departing from the spirit and scope thereof and, therefore, the invention is not to be limited except as defined in the appended claims.

I claim:

1. A process for the production of tubular casings comprising guiding a flexible sheet of a cellulosic material longitudinally to a forming position, said sheet being prepared from a starting material containing as an essential component a hydroxyalkyl cellulose ether and said sheet being plasticized with a minor amount of urea, continuously shaping the sheet into a tubular form with the longitudinal edges overlapped, and sealing the overlapped edges with an alkaline solution so as to form a sealed tubular casing, the urea content of the sheet coacting with the alkaline sealing solution to accelerate the sealing process.

2. Process of claim 1 wherein the sheet is formed by the coagulation of a cellulose ether-xanthate solution, the cellulose ether portion being hydroxyethyl cellulose.

3. Process of claim 1 wherein the urea is present in the sheet in an amount of 3–30% and the casing is sealed at a temperature of 10° C. or lower using an alkali concentration of not more than 1% by weight in the sealing solution.

4. In a process of sealing together portions of a flexible sheet comprising a water-insoluble, alkali-soluble cellulosic derivative by wetting areas of said sheet to be joined with a solution containing alkali and pressing said areas together while wet, the improvement comprising impregnating said sheet with from about 3 to 30% of urea based on the dry weight of said sheet prior to the wetting of areas of said sheet with said solution of alkali.

5. The improved process of claim 4 further characterized in that said solution contains from about 1 to 6% alkali.

6. The improved process of claim 5 further characterized in that prior to wetting with said alkaline solution a wetting agent and a polyhydric alcoholic plasticizer are applied to said sheet.

7. In a process as defined in claim 6 wherein the polyhydric alcohol is a compound of a group consisting of glycerine, ethylene glycol, diethylene glycol, sorbitol.

8. In a process as defined in claim 5, wetting the areas to be joined with an alkali solution having an alkali concentration no greater than about 1 percent, and chilling said areas wetted before the water of solution has substantially left said areas.

9. In a process of sealing together portions of a flexible sheet comprising a water-insoluble, alkali-soluble cellulosic derivative by wetting areas of said sheet to be joined with a solution containing alkali and pressing said areas together while still wet, the improvement comprising impregnating said sheet prior to wetting with said alkaline solution with at least 1.5% urea based on the dry weight of said sheet.

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