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**Stewart, Jr.**

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[54] **ACID-STEAM STERILIZATION**


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[63] Continuation of Ser. No. 70,803, Sept. 9, 1970, abandoned.

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[51] **Int. Cl.** ..... **B65b 7/28**

[58] **Field of Search** ..... 53/37, 22, 25, 11; 21/57, 21/79, 80; 99/182



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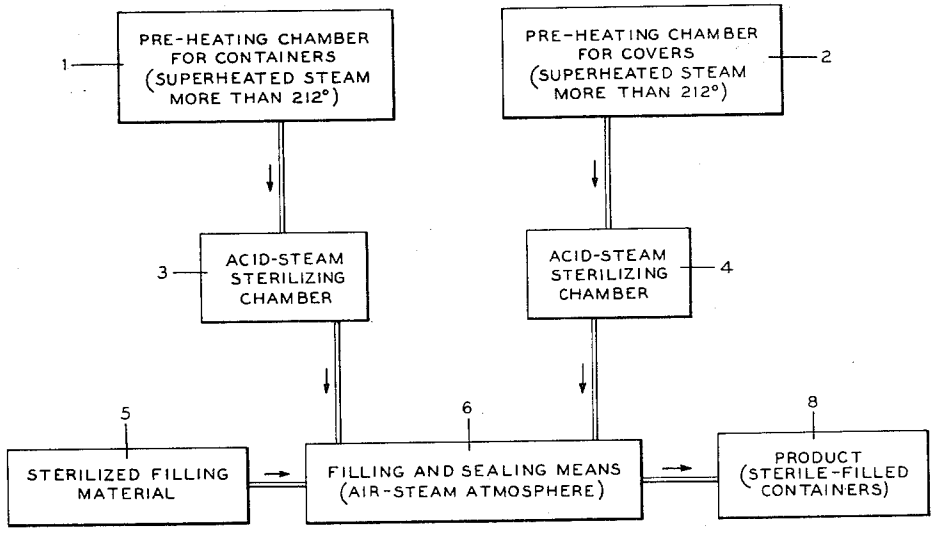
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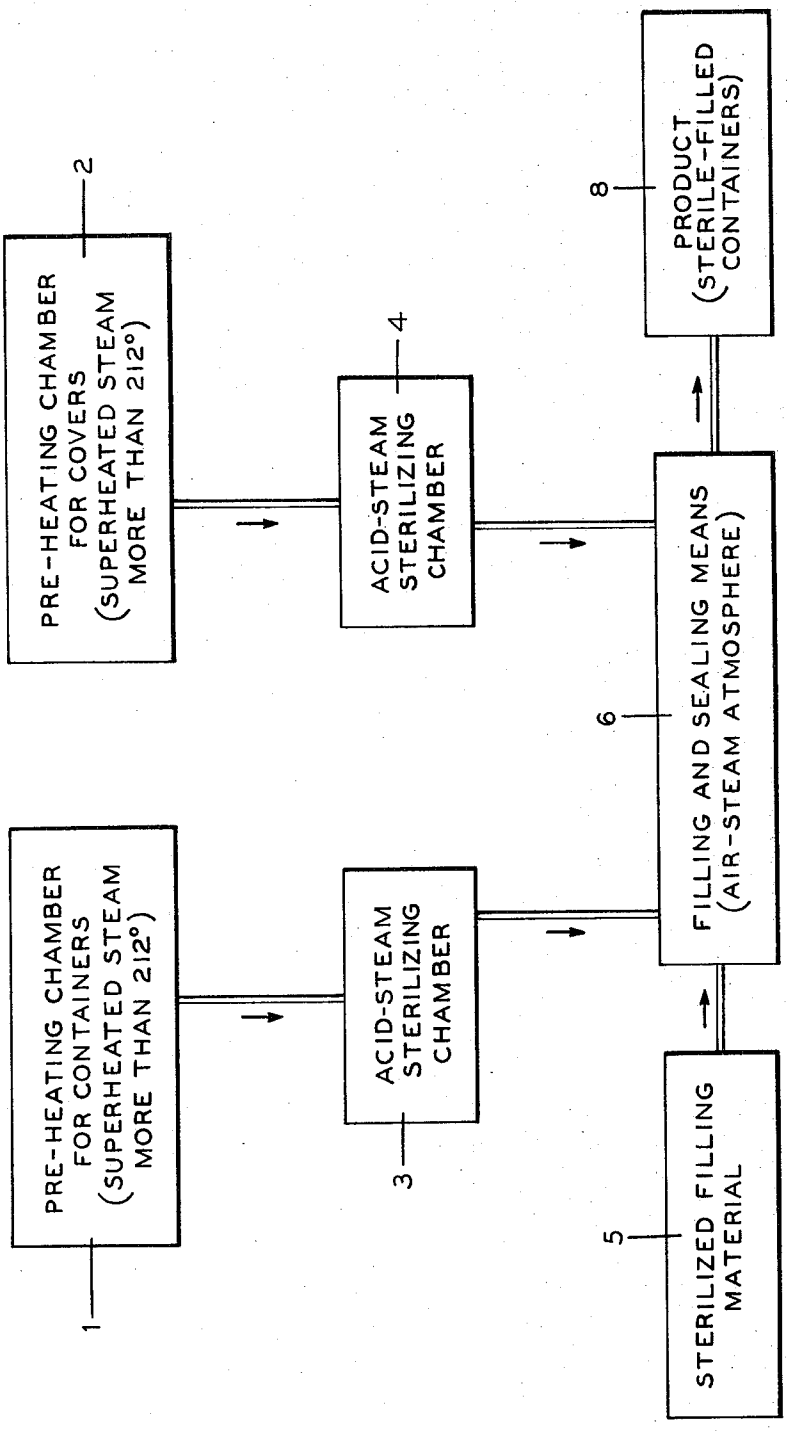


[57] **ABSTRACT**

A method for sterilization of containers. A plurality of open containers are heated by superheated steam to above 212°F. in a first heating chamber. The heated containers are then sterilized with an acid-steam mixture at a temperature of about 240° to 300°F. in a second heating chamber. The containers are filled and sealed in a sterile air-steam atmosphere immediately upon leaving said second heating chamber.


**1 Claim, 1 Drawing Figure**






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# ACID-STEAM STERILIZATION

This is a continuation of application Ser. No. 70,803, filed Sept. 9, 1970, now abandoned.

## BACKGROUND OF THE DISCLOSURE

The instant invention is directed to a method for sterilizing containers. More specifically, the instant invention is directed to a method for sterilizing containers in a two stage operation in which the second stage includes exposing the containers to an acid-steam mixture. Most specifically, the instant invention is directed to a method for sterilizing containers in a two stage operation in which the containers are first heated to a temperature of above 212°F. by superheated steam and thereafter sterilized in a second heating step in which the containers are exposed to an acid-steam sterilizing mixture.

In the prior art, various methods have been advanced for sterilizing containers. Included among these methods is the process of sterilization by the use of acidified steam. An atmosphere of acid-steam vapor destroys microorganisms at lower temperatures then an atmosphere of steam only. It is hypothesized that acid either weakens all or destroys many of the microorganisms present so that a lower temperature is required to completely destroy the remainder of the microorganisms present. Thus, the complete destruction of all microorganisms, or sterilization, can be accomplished at lower temperatures in the presence of acid. Therefore, steam sterilization is accomplished at lower temperatures, with all the attendant advantages thereof, when a small percentage of acid is added to the steam.

The method of acid-steam sterilization has been advanced, in the prior art, for sterilization of containers to be used in the packaging of a sterile product. Although the prior art methods provide advantages over steam sterilization methods as described above, they are subject to certain disadvantages which make their use less attractive.

In one process suggested by Shields et al (U.S. Pat. No. 3,139,323) an acid at a concentration of 0.36 to 3.6 percent by weight, in an acid-steam mixture, is used to sterilize containers. The temperature of the mixture is stated to be 150° to 212°F. The inventors suggest that subjecting articles to the mixture for a period of 10 seconds to 10 minutes is enough to sterilize the article. Although low temperature sterilization is an important advantage of acidified steam processes, at these relatively low temperatures a film of the acid remains on the surface of the container. Thus, the inventor admits that it is frequently necessary to rinse or heat vaporize the container prior to filling. Such an additional step is costly. Moreover, if heat vaporization is used, and as suggested by the inventor hydrochloric acid is employed as the acid, a constant boiling point mixture results. At atmospheric pressure, a constant boiling point hydrochloric acid-water mixture forms. This mixture boils at a temperature of 227.5°F. with a HCl concentration of 20.2 percent. At this high acid concentration corrosion of the container often results.

A second process suggested in the prior art, the patent to Martin (U.S. Pat. No. 3,180,740), describes a second acid-steam sterilization process. In it, Martin suggests wetting the container surface with an acid and then heating with steam at a temperature at a range of 212° to 270°F. Thus, Martin overcomes the low temperature disadvantage of the Shields et al method in so



far as the Martin process requires an acid-steam mixture temperature in excess of 212°F. for article sterilization. Although the Martin sterilization method occurs at higher temperatures it does not solve the corrosion problem discussed above. As the aqueous acid solution on the container surfaces is heated, the acid solution on the container surface is concentrated due to the formation of a constant boiling point mixture. As stated above, commonly used hydrochloric acid boils at the high acid concentration of 20.2 percent. This, of course, can result in corrosion of the container surfaces. If rinsing is substituted for acid boiling, the additional cost of rinsing similarly makes the Martin process less attractive.

Thus, the prior art methods employing acid-steam for sterilization are subject to the problem of the presence of acid solution on the container surfaces. This, in turn, results in corrosion of the containers, or alternatively to an additional rinsing step. This and other disadvantages are overcome by the method of the instant invention which is described in greater detail hereinafter.

## SUMMARY OF THE INVENTION

The instant invention is directed to a process for sterilization of containers by acid-steam contact in which no acid is condensed. Therefore, the instant invention is directed to a process of acid-steam sterilization in which the problem of container surface corrosion does not arise. This is accomplished by preheating the containers above the temperature at which the acid-steam mixture condenses prior to exposure of the containers to said acid-steam mixture atmosphere.

In accordance with the instant invention, a method is provided for sterilizing containers in which a plurality of containers are heated by superheated steam to a temperature in excess of 212°F. in a first heating chamber. The heated containers are then sterilized in an acid-steam atmosphere at a temperature in excess of the acid-steam condensation temperature in a second heating chamber. They are thereafter immediately filled and sealed in a sterile atmosphere.

## BRIEF DESCRIPTION OF THE DRAWING

The instant invention may be better understood by the accompanying drawing which is a block diagram of a preferred embodiment of the method of the instant invention.

## DETAILED DESCRIPTION

Turning to FIG. 1 in detail, the pre-heating or first heating stage is directed in steps 1 and 2. Step 1 illustrates the pre-heating of the containers. It should be understood that the containers include uncovered cans, receptacles and the like. The containers are heated in the first heating stage 1 to a temperature in excess of 212°F. More preferably, the containers are heated in stage 1 to a temperature in the range of about 225° to 240°F. In a preferred embodiment the heating medium is superheated steam. Steam is a preferred method for pre-heating of containers. As between saturated steam and superheated steam, superheated steam is preferred since it permits the attainment of higher temperatures at lower pressures. This, in turn, not only insures the structural integrity of the containers, but also eliminates the necessity of a pressurized chamber, which is required if temperatures appreciably in excess of 212°F. are required and saturated steam is used. It

should be appreciated that if a chamber designed to withstand higher pressures is available and the containers themselves are of sufficient strength to withstand pressures in excess of atmospheric saturated steam may be used. It should be further appreciated that other heating means, other than steam, may be substituted as long as the containers leave the first heating stage at a temperature of more than 212°F. and preferably 225°F. to 240°F.

In one preferred embodiment, the containers are disposed on a container moving means such as a porous conveyor belt in an upside down configuration. That is, the containers are placed on the porous belt with open end rims of the containers contacting the belt. In this way, any condensate that may form in the container can drain to the open end of the container and drop to the bottom of the heating chamber through the pores of the porous belt.

In the preferred embodiment wherein the containers are disposed on porous belt and are heated by steam, the steam, which is preferably superheated, enters the chamber so as to heat the containers by countercurrent convective heat transfer. Thus, the steam enters the first heating stage chamber at the container outlet end thereof and leaves through a steam exiting means located at the container inlet end of the chamber. The expended steam, which exits through the steam exiting means may be reheated and recycled for reuse. Alternatively, fresh steam may be continually employed.

The heated containers leave the pre-heating stage **1** at a temperature of at least approximately 212°F. This is insured by employing countercurrent heat transfer means in stage **16.** As the containers leave the first heating chamber, they are heated by steam, just entering the chamber. The already partially heated containers are thus heated to a temperature approaching that of the incoming steam which enters at a temperature in excess of 212°F.

The lids, which are to be disposed on and sealed to the open containers, as will be described in greater detail hereinafter, are also treated in a first heating or preheating stage. This is depicted in the drawing as heating stage **2.** In stage **2,** the lids, which may include a sealing membrane, as well as an outer cover, are treated in a manner exactly analogous to the treatment afforded the containers in heating stage **1.** It should be understood that when the word lid is used it is descriptive of a membrane also. Thus, in a preferred embodiment a plurality of lids are heated by the countercurrents convective heat transfer with superheated steam as the heating medium. Again, saturated steam or even heating means other than steam may be substituted. Since, the pre-heating treatment of the lids is the same as the pre-heating treatment of the containers, they may be treated in the same chamber. In this case, a plurality of containers are first heated, followed by a plurality of lids. Alternatively, a container followed by a lid may be disposed on the porous conveyor belt. Of course, two separate chambers, one for containers and one for lids, as suggested in the drawings as stages **1** and **2,** respectively, may likewise be employed.

Returning now to the treatment of the containers, they leave the pre-heating stage **1** at a temperature of at least 212°F. and preferably at a temperature of about 225°F. to 240°F. The heated containers immediately enter an acid-steam sterilizing or second heating stage **3.** The sterilizing stage **3** comprises another chamber, which in a preferred embodiment, is again provided with a container moving means. Typical of such means is again a porous conveyor belt. The containers are again preferably disposed on the belt in an inverted configuration with opened end of the containers in contact with the belt. This positioning is a redundant safety procedure, as will be described in greater detail hereinafter. The containers on the belt are heated by an acid-steam gaseous mixture which flows countercurrently to the movement of the plurality of containers stationed on the moving porous belt. Thus, the advantages of countercurrent convective heat transfer are again derived.

The unique acid-steam gaseous mixture provides the sterilizing means in the method of the instant invention. A small amount of acid, which in a preferred embodiment is hydrochloric acid or acetic acid is added to steam to provide the sterilizing mixture. It should be appreciated that other acids may be substituted for the acids mentioned above, but usually hydrochloric or acetic acid is employed. Independent of the acid utilized, enough acid is added to the steam so that the gaseous mixture has a pH in the range of about 2.8 to 3.2, as measured in the condensate, after the steam has left the chamber and has been cooled. In the preferred embodiments wherein hydrochloric or asetic acid is used, 0.06 percent down to 0.023 percent by weight of hydrochloric acid, or 0.90 percent down to 0.19 percent by weight of acetic acid is added to steam to provide a pH in the preferred range of 2.8 to 3.2, respectively. It should be understood that the pH of the gaseous mixture may be outside the range indicated. However, it has been found that sterilization, is optimized under these conditions.

The steam employed is preferably superheated for the reasons given above. Thus, it is possible to use a saturated steam-acid mixture if the chamber and containers are properly designed to withstand the increased pressures of saturated steam. However, in the sterilizing stage **3,** there is an additional reason for employing superheated steam. This reason, which will be discussed below, makes it even more preferable to employ an acid-superheated steam mixture.

Whether the steam is saturated or superheated, the acid-steam mixture enters the sterilizing chamber at a temperature range in excess of the acid-steam condensation temperature. Preferably, this temperature ranges from about 240° to 300°F. At these temperatures, and in the presence of acid, and in the concentration range noted above, the containers are sterilized. It has been found that a residence time of 10 to 25 minutes is required to sterilize containers in sterilizing stage **3** at the lower end of the sterilizing temperature range, that is, about 240°F. At the upper sterilizing temperature range of approximately 300°F., the residence time is noticeably decreased to a period of about 1 to 5 minutes and preferably 3.5 minutes. Thus, the containers have a residence time of about 1 to 25 minutes, when an acid-steam mixture having a condensate pH of about 2.8 to 3.2 and a temperature of about 240° to 300°F. is used as the sterilizing medium. The time-temperature combination chosen by the sterilizer is usually a function of container material. Thus, a metal container will usually withstand higher temperatures then a plastic container. Therefore, metal containers are usually sterilized at higher temperatures for shorter times than plastic containers.

The containers exiting the first heating stage **1**, as stated above, are heated to a temperature of at least 212°F. Hence, there is no possibility that cold container surfaces will condense any of the acid-steam mixture in sterilizing stage **3** at atmospheric pressure. Acid-steam condensation can cause container corrosion, as discussed above. Thus, the method of the instant invention eliminates the problem of corrosion from acid-steam container sterilization. It should be appreciated, from the above, that in order to insure non-condensation during sterilization that the entrance temperature of the containers into the sterilization stage **3** must be greater than 212°F. if the acid-steam mixture pressure is higher than atmospheric. Since the preferred entrance temperature is about 225° to 240°F. this should not be a problem even if the steam is only slightly superheated. Of course, the more superheated the steam in the acid-steam mixture the more remote the possibility of condensation.

A final precaution, mentioned above, is also employed to guard against the remote possibility of acid condensation. That is the disposition of the open containers in an upside down configuration on the belt. If any condensation does occur, it is probable that the condensate will run out the open end prior to the container leaving the chamber.

An analogous sterilizing step occurs in sterilizing stage **4.** In stage **4** the lids are sterilized under the same conditions as discussed above in regard to the operation of stage **3.** The only distinguishing feature between lid sterilization and container sterilization is that the lids cannot be disposed on the porous belt in an upside down position. A preferred design provides for designing the conveyor belt so that the lids are disposed in an upright position. This preferred configuration not only permits condensate removal but better exposes the lid surfaces to the sterilizing acid-steam mixture. Because of the similarity of operating conditions in stages **3** and **4,** in one preferred embodiment, sterilization of the lids and the containers occurs in the same chamber. The use of a single chamber is usually preferred in those cases where the operation of the pre-heating stages **1** and **2** is accomplished in a single pre-heating chamber.

Following sterilization in stages **3** and **4,** which may occur in one or two chambers, the sterilized lids and containers are placed in a chamber which constitutes the filling and sealing stage **6.** Previously sterilized filling material **5** is placed into the open sterilized containers, while the containers are in the filling and sealing stage **6,** by filling means well known in the art. After filling of each container to a point below the top of the open end, a lid is placed on the open end and sealed thereto. The filling and sealing chamber is presterilized and maintained sterile by a sterile steam-air atmosphere maintained at a pressure of about 2 to 6 inches of water above atmospheric pressure. This atmosphere permits vacuum sealing. The steam-air atmosphere fills the headspace between the top surface of the sterile material and the lid. After the sealed containers leave the filling and sealing stage **6,** they are cooled. This results in condensation of the steam in the headspace and the resultant formation of a vacuum. The final product, in sealed containers is designated in the drawing by reference numeral **8.**

It should be appreciated that the above-described preferred embodiment of the method of the instant invention is meant to be illustrative only. The foregoing specification and drawing will make apparent various modifications which are within the contemplation of the instant invention. Thus, the scope of the invention should be limited only by the appended claims.

What is claimed is:

**1.** A method for aseptic canning of a sterile product comprising the steps, in seriatim, of:

a. first preheating a plurality of open containers and sealing lids to a temperature of about 225° to 240°F. under substantially atmospheric pressure; and then

b. sterilizing said heated containers and lids by exposing them to an acid-steam mixture maintained at substantially atmospheric pressure and at a temperature in the range of 240° to 300°F. for a period of about 1 to 25 minutes, said mixture having a pH of about 2.8 to 3.2 as measured in the condensate of said acid-steam mixture; and thereafter

c. canning said sterile product in a sterile air-steam atmosphere maintained at a pressure 2 to 6 inches of water above atmospheric pressure by filling said open containers with said sterile product, covering said containers with said lids and thereafter sealing said lids to said containers.

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