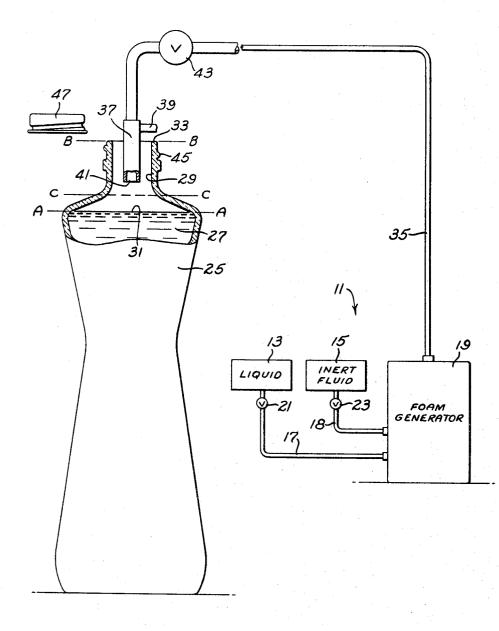
## Oct. 15, 1968

# W. P. GIBBLE

3,406,080

PACKAGING OF SALAD OILS AND THE LIKE Filed April 30, 1965



INVENTOR.

WALTER P. GIBBLE

BY HIS ATTORNEYS. HARRIS, KIECH, RUSSELL & KERN 5

45

1

### 3,406,080

PACKAGING OF SALAD OILS AND THE LIKE Walter P. Gibble, La Habra, Calif., assignor to Hunt Foods and Industries, Inc., Fullerton, Calif., a corporation of Delaware

Filed Apr. 30, 1965, Ser. No. 452,167 7 Claims. (Cl. 99-171)

#### ABSTRACT OF THE DISCLOSURE

Air or other headspace gas is displaced from the headspace of an open-top container by a foam having a component of inert gas and a component that is at least partially frozen. After the container is closed the foam is disintegrated leaving the headspace filled with inert gas. 15

This application relates to a novel method of packaging and more particularly to a novel method of removing air from the headspace of a container by injecting a foam 20 therein.

Many products deteriorate in the presence of air. It is necessary, therefore, to purge the air from the headspace of the containers used to package such products to prevent deterioration thereof. One prior art method of air 25 removal was to inject a foam into the headspace to displace the air therefrom. Such foams consist of an inert gas and a liquid which are supplied to the headspace at or near ambient temperature.

It is desirable for the packaging of liquid products, 30 that the liquid component of the foam be the same material as the product. Difficulty arises, however, because often the liquid product does not make a stable foam, thereby reducing the efficiency of the oxygen removal. In an attempt to overcome this problem, prior art meth-35 ods have added an emulsifying agent to the inert gas and the liquid product in an attempt to produce a more stable foam. This is unsatisfactory because the addition of an emulsifying agent complicates the process and may have an undesirable effect on the product packaged. 40

An object of this invention is to provide a simplified process for producing a foam-like mass, herein referred to merely as a foam, and for discharging a quantity thereof into the headspace of a container to displace the headspace gas therefrom.

Another object of this invention is to eliminate the need for an emulsifying agent in producing a stable foam for use in packaging.

Another object of this invention is to produce a stable foam for use in the headspace of a container by entraining an inert gas in a matrix that is at least partially in solid phase to produce a foam having an inert gas component and an at least partially solid phase component. The dispersed phase of the foam is gaseous and what corresponds to the continuous phase of a conventional foam 55 is, in the invention, essentially a multitude of minute frozen masses of the liquid, with or without some of the latter remaining in liquid state.

A primary object of this invention is to produce a stable foam for use in packaging by mixing an inert fluid 60 and a liquid while at least partially freezing the liquid to produce a pourable foam having an inert gas component and another component that is at least partially frozen. According to another object, the liquid used to produce the foam may be a quantity of the product which is packaged in the container. Freezing of the liquid allows production of a stable foam without an emulsifying agent even though the liquid would not make a stable foam at ambient temperature. As no emulsifying agent is needed, the product purity is improved and the process is simplified. This is particularly important when packaging food products such as salad oils. 2

When such a foam is in the headspace, it provides a thick blanket which eliminates splashing of the liquid product from the container prior to capping. The foam of the invention is very stable for a period of a few minutes during which time it will withstand considerable vibration without deterioration. After the passage of several minutes, however, the frozen component thaws or melts causing the foam to disintegrate into the liquid product and an inert headspace gas. The resulting liquid product joins the product in the container, leaving the residual headspace filled with the inert gas.

Another object of this invention is to provide a foam for use in packaging by intimately mixing a quantity of the liquid product to be packaged with a cold inert fluid to at least partially freeze such quantity of product and to substantially simultaneously foam the inert fluid and the quantity of product to produce a foam having an inert gas component and an at least partially frozen product component. Thus, the freezing and foaming may take place substantially simultaneously and in the same piece of equipment. As the foam consists of a cold gas in an at least partially frozen liquid matrix, the density thereof is much greater than the density of the air or other headspace gas initially in the headspace. Accordingly, it will more readily displace the air or headspace gas from the headspace than would a lighter or less dense foam.

Another object of this invention is to produce a foam utilizing an inert liquid such as liquefied nitrogen for the inert fluid, the liquefied nitrogen becoming a gas by absorbing heat from the quantity of product during the freezing of the latter. Still another object of this invention is to produce a stable foam for use in bottling salad oils by vigorously mixing liquid nitrogen and liquid salad oil while at least partially freezing the salad oil to produce a stable foam having an inert gas component derived from the liquid nitrogen and another component that is at least partially frozen.

A further object is to produce a viscous but pourable foam for use in packaging or otherwise, the foam having an inert gas component and a solid phase component of minute solid phase particles. The minute solid particles produce a foam that can be poured into the headspace to displace air or headspace gas therefrom. It is an object of this invention to produce such a foam by stirring an inert fluid and a quantity of product during the freezing of the latter.

An object of this invention is to control the fill heights of containers for liquid products by varying the ratio of product to inert gas in the foam which is injected into the headspace thereof. By at least partially freezing the liquid component of the foam, the percent of inert gas by volume may vary over a wide range such as from about 20% inert gas to well above 50% inert gas.

The invention, both as to its organization and method of operation, together with further objects and advantages thereof, may best be understood by reference to the following description taken in connection with the accompanying drawing which illustrates diagrammatically the equipment which may be used in practicing the process of this invention.

Referring to the drawing, a headspace purging system 11 includes a liquid container 13 and an inert fluid container 15 connected by separate conduit means 17 and 18, respectively, to a foam generator 19. The liquid container 13 contains a quantity of liquid which is to form one component of the foam generated in the foam generator 19. Preferably such liquid is the same product which is being packaged or a component thereof. The inert fluid container 15 contains an inert fluid, preferably liquid nitrogen or cold nitrogen gas at a temperature of  $-100^{\circ}$ C. to  $-190^{\circ}$  C., with a preference for a temperature of

about  $-145^{\circ}$  C. to  $-155^{\circ}$  C. when making a foam useful in packaging liquids such as salad oils where a temperature of about  $-150^{\circ}$  C. will be found satisfactory. Other liquefied (e.g., octafluorocyclobutene) or cold inert fluids can be used, e.g., carbon dioxide, the noble gases 5 such as helium, neon, argon, etc., or nitrous oxide. Nitrogen gas is particularly advantageous. For nonfood packaging, butane, isobutane, and propane may be used as inert gases.

A pair of control valves 21 and 23 meters and controls 10 the flow of liquid and inert fluid, respectively, into the foam generator 19. The liquid and inert fluid are fed to the foam generator through the separate conduits to avoid mixing thereof and freezing of the liquid.

The foam generator 19 is preferably a high intensity 15 mixing device employing rotating blades, shear spaces, fluid impingement, etc., with or without recirculation to bring the liquid and the inert chilling fluid into intimate contact to effect the desired degree of freezing of the liquid and the production of the foam. In a small scale 20 operation a conventional high-speed laboratory-size electric blender can be used, e.g., a commercially available unit known as a "Waring Blendor" can be used. However, other types of foam generators may be used. For example, the foam may be produced by continuous ultra- 25 sonic homogenization, in-line pressure gas sparging, high speed pressurized agitation, or restricting the supply of components to a high speed centrifugal pump.

In the foam generator 19 the liquid and the inert chilling fluid are thoroughly mixed. The inert fluid is supplied 30 at a temperature below the freezing point of the liquid so that this mixing causes the liquid in the foam generator 19 to become at least partially frozen. If the inert fluid is supplied as a liquefied gas, the relatively warm liquid heats the inert fluid, causing it to become a gas. Substan- 35 tially simultaneously with the freezing of the liquid, the foam generator produces a foam having an inert gas component entrained in a matrix of an at least partially frozen component.

An ordinary gas-in-solid foam-type system, e.g., a 40 sponge, will not flow and cannot be poured. An important feature of this invention is the production of a foam having an inert gas component and an at least partially frozen component which is pourable. This is accomplished by forming the external phase of minute at least partially 45frozen particles that are solid. Thus, the foam includes a matrix of minute substantially solid phase particles having entrained therein an inert gas. Such a foam, while viscous, is pourable. The foam generator 19 produces such a foam by vigorously stirring the liquid and the 50 inert fluid, it being understood that the liquid will commence freezing upon contact with the cold inert fluid. Such foam will preferably consist substantially exclusively of an inert gas and a substantially frozen component. Some of the liquid may also be present in the liquid phase 55 in this substantially frozen component.

The drawing shows a container 25, the interior of which has been substantially completely filled with a product 27 leaving a small headspace 29 filled with air. The headspace 29 is defined by the interior wall of the container 60 25 between the product surface 31 at an initial level A-A and an open top end 33 of the container 25.

In the embodiment illustrated, the container 25 is a glass bottle; however, no limitation is intended thereby as the invention may be used with containers of other ma- 65 terials. The product 27 is exemplified as a liquid such as salad oil which will tend to deteriorate or become unstable in the presence of air. The process described herein may be used with various products both liquid and solid, edible and nonedible.

A conduit 35 conducts foam from the foam generator 19 to a filling tube 37 which is liftable or slideable axially on the outlet end of the conduit. The filling tube 37 has a handle 39 for adjusting its elevation relative to the upper product surface 31 and a downwardly directed dis. 75 simple requiring only two components and a single foam

charge port 41 which is shown as positioned in the headspace 29 above the upper product surface. Alternatively, the discharge port may extend laterally through the side wall of the filling tube 37. A control valve 43 controls the flow of foam to the headspace 29.

It is preferred that a portion of the tube 37 and the discharge port 41 be positioned in the headspace 29 with the port above the upper product surface 31. In other instances the discharge port 41 may even be positioned out of the headspace slightly above the open top end 33 if the foam is of a density greater than the ambient air. In either instance the tube 37 may be positioned axially within the headspace or off-center adjacent the interior wall of the container. The vertical space between the upper product surface 31 and the discharge port 41 may be varied to produce a headspace of the volume desired. It should be understood that various filling line techniques may be used for injecting the foam into the headspace 29 and that the vertically movable filling tube 37 is merely shown by way of illustration.

Discharging a stream of the heavy pourable foam into into the headspace 29 readily displaces the air therefrom through the open top of the container. The foam has a greater density than air at ambient temperature in the headspace 29, and is very effective in displacing air from the headspace 29.

As the filling tube 37 is withdrawn from the headspace, the foam may continue to discharge therefrom to fill the small volume of the headspace previously occupied by the walls of the filling tube. The headspace 29 is at this time filled with the foam to a level B-B at or near the top end 33 of the container. The viscous foam forms a thick blanket in the headspace which eliminates splashing of the liquid product 27 as the container is moved to the capping station. During this interval prior to capping, the high density and high stability of the foam prevent the entry of air into the headspace 29.

The upper end of the container 25 is provided with screwthreads 45 onto which a cap 47 may be secured either manually or by suitable capping apparatus. If desired, the cap 47 may be laved with an inert gas such as nitrogen immediately prior to capping but this is usually not necessary. Of course, various other means may be employed to close the container 25. When capped, the headspace 29 is substantially filled by the foam.

For a short period of time, the foam is very stable and will withstand considerable vibration without deteriorating. After capping and after the passage of a short period of time, e.g., at least 10 minutes under average conditions, the frozen component of the foam is melted by heat conduction from the product and/or heat transmitted through the container and its cap from the ambient air or from some other source of heat. This causes the foam to disintegrate into the liquid product and an inert headspace gas. The resulting increment of liquid product joins the main body thereof and causes the surface thereof to rise to the desired ultimate level C-C. Thus when the frozen component is a quantity of the product 27 or a component thereof, the composition of the foam may be utilized to control the fill height of the container. As the relative amounts of inert gas and product in the foam may be varied over a wide range, it follows that the foam process of the invention is versatile in permitting any desired initial filling of the container to a desired initial level A-A.

An important feature of this invention is the production of a stable foam consisting essentially of an inert gas and a portion of the product that is at least partially 70 frozen, the emulsifying agent needed by prior art methods being completely eliminated. It is also important that this foam is rendered pourable by producing the solid phase component from minute solid phase particles. The method of foam production described herein is very

generator. The dense stable foam produced thereby is very effective in displacing and excluding air from the headspace of a container.

Many changes, modifications, and substitutions may be made by one having ordinary skill in the art without de- 5

parting from the spirit and scope of this invention. What is claimed is:

1. A process for packaging a liquid product in an open topped container, including the steps of:

- at least partially freezing a quantity of said liquid 10 product by intimately mixing said quantity of said liquid product and a liquefied inert gas to produce a pourable foam having said inert gas entrained in a matrix of minute substantially solid phase product particles; 15
- substantially completely filling said container with said liquid product while leaving a small headspace filled with a headspace gas;
- discharging a quantity of said pourable foam downwardly into said headspace to displace the headspace 20 gas therefrom and fill the headspace with said foam; and
- closing the top of said container to entrap said foam in said headspace.

2. A process for packaging salad oil in an open topped 25 container, including the steps of:

providing cold nitrogen below the freezing temperature of said salad oil;

- vigorously mixing said cold nitrogen and a quantity of said salad oil at an initial temperature higher 30 than that of said cold nitrogen to produce a pourable foam having a matrix of minute substantially frozen salad oil particles containing said nitrogen in gaseous form;
- substantially completely filling said container with 35 said salad oil while leaving a small headspace filled with air;
- discharging a quantity of said foam directly into said headspace to displace the air therefrom; and
- closing the top of said container to entrap said foam 40 in said headspace.

3. A process for packaging a liquid product in an open-topped container, including the steps of:

- producing a low-temperature foam having an inert gas component and a component comprising a liquid that 45 is at least partially frozen, said foam being produced by mixing with said liquid said inert gas in liquefied state;
- substantially completely filling the interior of said container with said liquid product while leaving a 50 small headspace above the product level filled with a headspace gas;
- introducing a quantity of said foam directly into said headspace to displace therefrom the headspace gas and fill the headspace with said foam; and 55
- closing the top of said container to entrap said foam in said headspace.

4. A process as defined in claim 3 including the additional step of exposing the closed container to an ambient 6

temperature sufficient to disintegrate said foam and produce an inert headspace gas filling said headspace and a liquid that enters and becomes associated with said product.

5. A process as defined in claim 4 in which said at least partially frozen liquid of said foam comprises a small portion of the same liquid as the product whereby said disintegration of said foam in said headspace produces a liquid identical with the product liquid in said container dropping to the product liquid and joining the body thereof in the container thereby raising the product level therein.

6. A process for packaging a liquid product in an open-topped container, said product being liquid at ambient temperature, said process including the steps of:

- intimately mixing a small quantity of said liquid product with a liquefied inert gas to form a low-temperature foam having an inert gas component and a component comprising said small quantity of said liquid product that is at least partially frozen;
- substantially completely filling the interior of said container with said liquid product while leaving a small headspace above the product level filled with a headspace gas;
- introducing a quantity of said low-temperature foam directly and exclusively into said headspace to displace therefrom the headspace gas and fill the headspace with said foam, the temperature of the foam thus introduced being substantially less than ambient temperature, the foam thus introduced containing said at least partially frozen component;
- closing the top of said container to entrap said lowtemperature foam in said headspace; and
- subsequently bringing the closed container and the liquid product therein to ambient temperature to thaw the at least partially frozen component of the foam and release (1) said small portion of said liquid to drop to and become associated with said liquid product in the container thereby raising the product level therein and reducing the volume of the headspace and (2) the inert gas component of the foam to form an inert headspace gas now filling the headspace of reduced volume.
  - to form an inert headspace gas now filling the headspace of reduced volume.

7. A process as defined in claim 6 in which the volume percent of inert gas in said low-temperature foam is from about 20% up to at least about 50%.

#### References Cited

#### UNITED STATES PATENTS

2,433,071	12/1947	Stevenson	99-189
2,583,697	1/1952	Hendry	99-189
3,068,105	12/1962	Morrisson	99-192

RAYMOND N. JONES, Primary Examiner.

E. A. MILLER, Assistant Examiner.

### **U.S. DEPARTMENT OF COMMERCE**

### **PATENT OFFICE**

Washington, D.C. 20231

# UNITED STATES PATENT OFFICE CERTIFICATE OF CORRECTION

Patent No. 3,406,080

October 15, 1968

### Walter P. Gibble

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 3, line 26, before "gas" insert -- nitrogen --.

Signed and sealed this 10th day of March 1970.

(SEAL) Attest:

Edward M. Fletcher, Jr. Attesting Officer

### WILLIAM E. SCHUYLER, JR.

**Commissioner of Patents** 

ŝ,