United States Patent [19]

Bazett

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[45] **Feb. 13, 1973**

[54] **METHOD OF STABILIZING THERMOPLASTIC CONTAINERS**

[75] Inventor: **Patrick Seymour Bazett,** London, Ontario, Canada

[73] Assignee: **Kemp Products Limited,** Ontario, Canada

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[52] **U.S. Cl.** **264/25,** 264/235, 264/346

[51] **Int. Cl.** **B29d 23/00**

[58] **Field of Search**.....264/22, 25, 26, 235, 346

[56] **References Cited**

UNITED STATES PATENTS

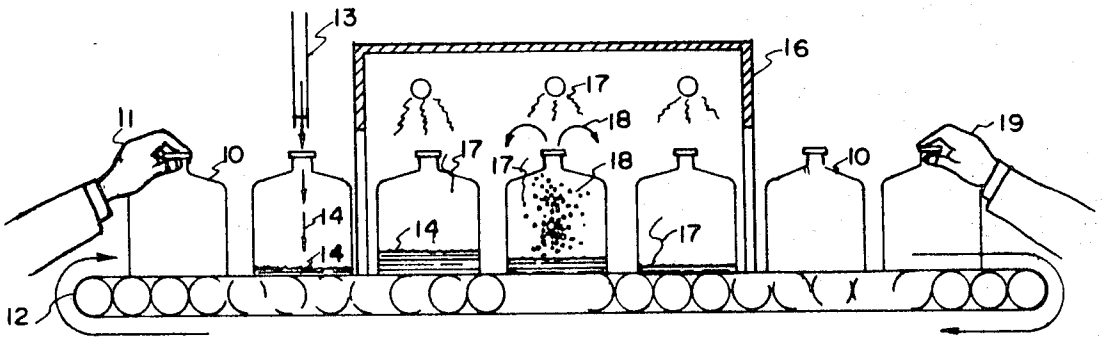
3,317,642	5/1967	Bailey	264/235 X
2,465,130	3/1949	Story	264/26
3,010,157	11/1961	Cizek	264/25
3,511,899	5/1970	Miller	264/26

Primary Examiner—Robert F. White
Assistant Examiner—Richard R. Kucia
Attorney—Philip T. Mitches

[57] **ABSTRACT**

Thermoplastic containers, particularly polyethylene, can be stabilized with desired or required control limit on volume and shape, such that they are capable of being used at temperature ranges in excess of 110°F. without appreciable or significant loss of shape or capacity. This is achieved by placing a small amount of liquid such as water within the bottle and subjecting the bottle to a microwave radiant energy field such that the liquid (water) absorbs the radiant energy and boils off into steam. The vapor generated would appear to be converted (after all water has evaporated) into superheated vapor which scours the interior of the bottle causing it to shrink and to stabilize such that under subsequent elevated heat environments (service temperatures) it is essentially stable in volume and shape.

7 Claims, 9 Drawing Figures



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SHEET 1 OF 4

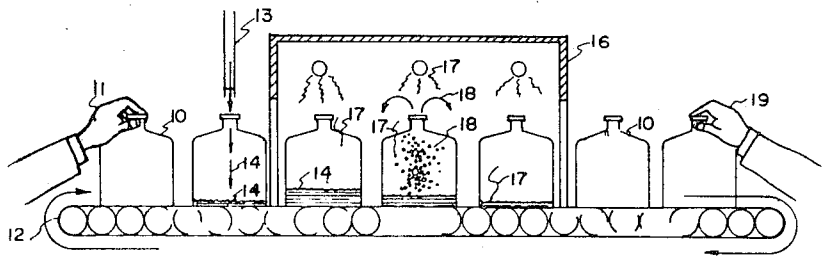


FIG 1

INVENTOR:

Patrick S. Bazett

AGENT:

Philip T. Hitchcock

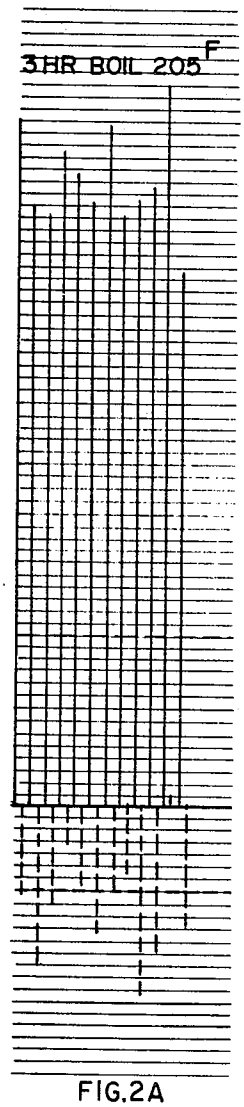


FIG.2A

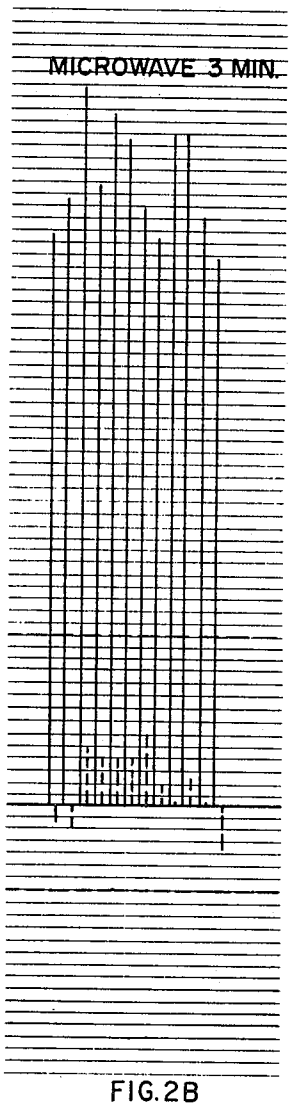


FIG.2B

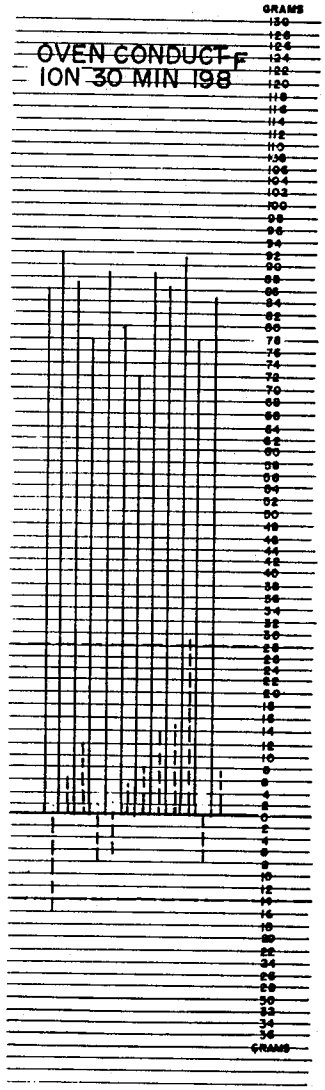
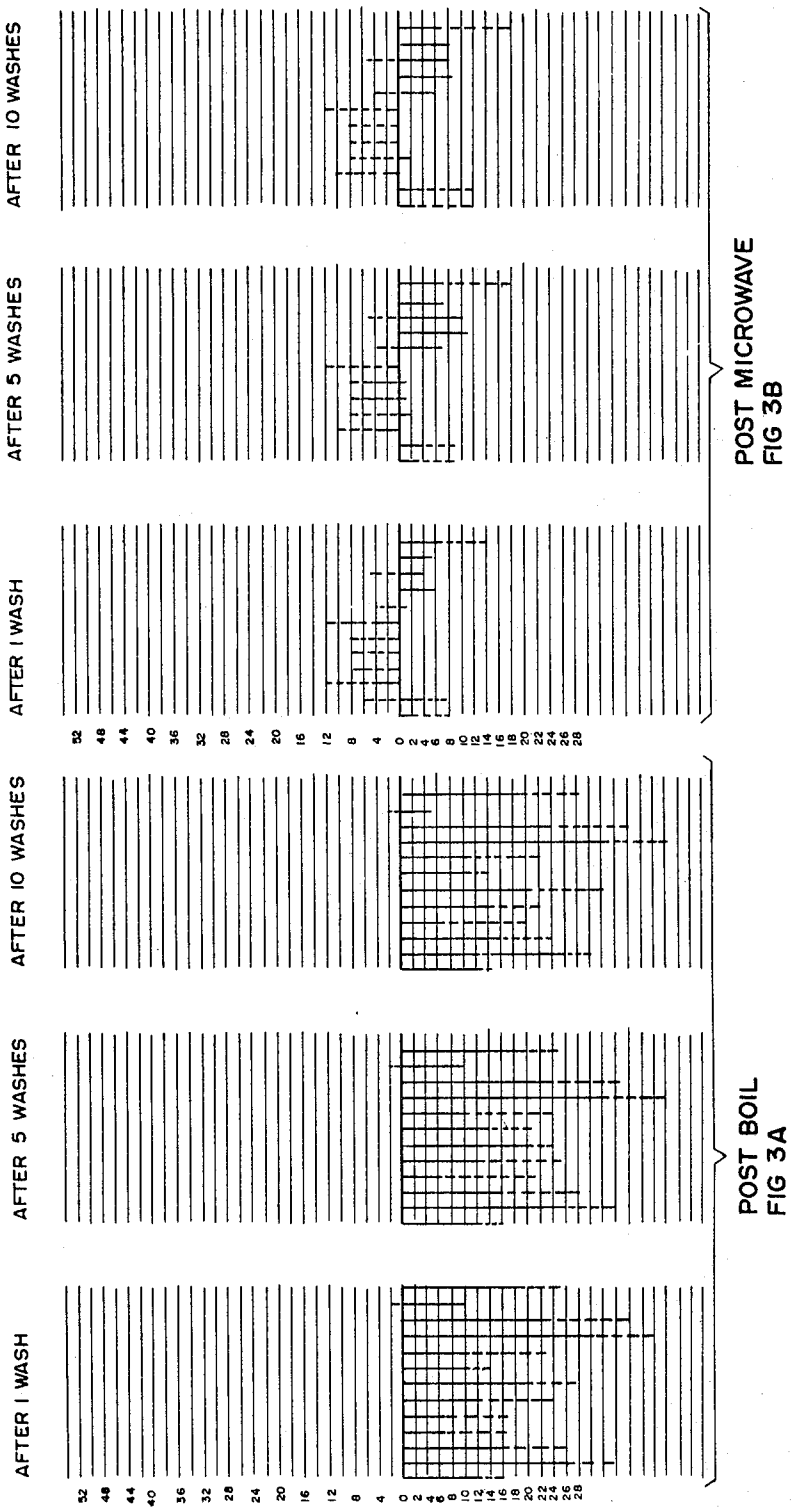


FIG.2C

VOLUME IN GRAMS BEFORE SHRINKING ————————

VOLUME IN GRAMS AFTER SHRINKING — — — — — — — —

INVENTOR:
Patrick J. Doggett
AGENT:
Philip T. Liebig


AFTER 10 WASHES
AFTER 5 WASHES
AFTER 1 WASH
POST MICROWAVE
FIG 3B
AFTER 10 WASHES
AFTER 5 WASHES
AFTER 1 WASH
POST BOIL
FIG 3A
32 48 44 40 36 32 28 24 20 16 12 8 4 0 2 4 6 8 10 12 14 16 18 20 22 24 26 28

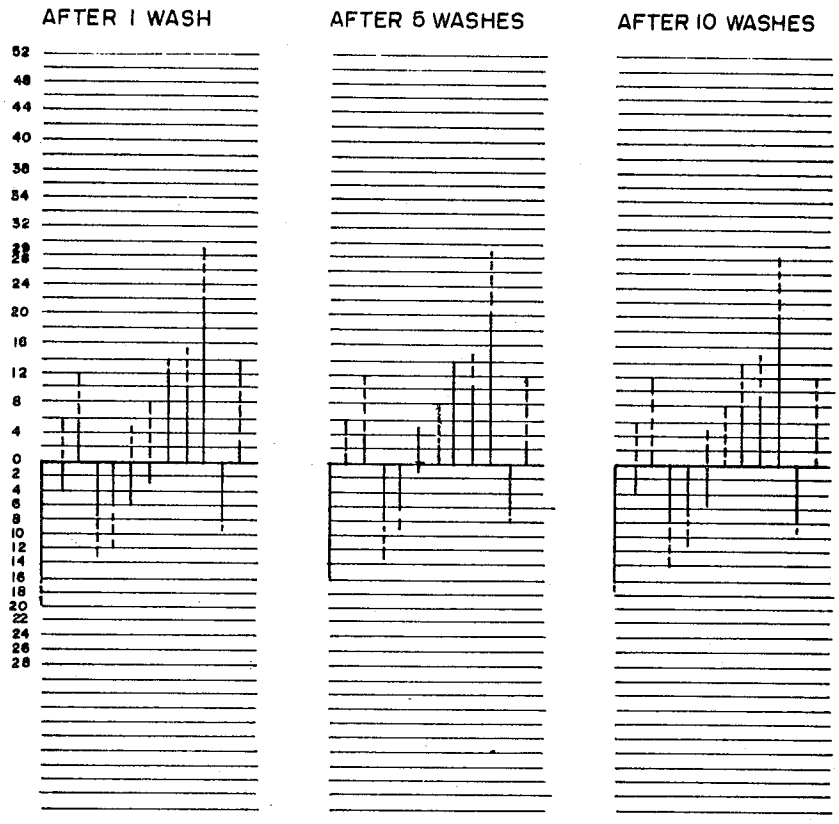


FIG 3 POST OVEN CONDUCTION

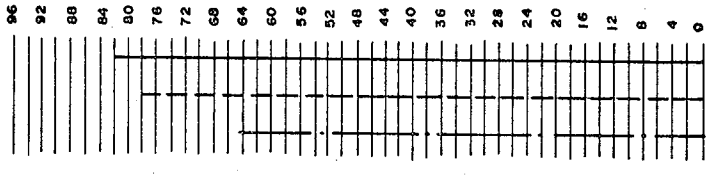


FIG 4 AGGREGATE LOSS IN GRAMS OF CAPACITY AFTER 10 WASHING CYCLES

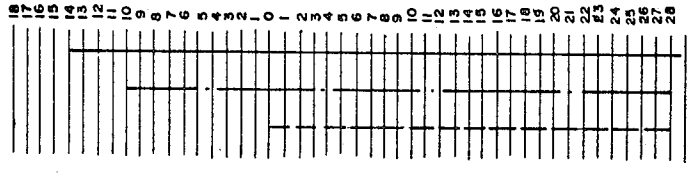


FIG 5 COMPARITIVE CONTROL LEVEL RANGE SHOWING HI-LO VARIATIONS OF CAPACITY (IN GRAMS) BETWEEN 0-10 WASHING CYCLES

INVENTOR
Robert S. Dyett
AGENT
W. J. Trick

METHOD OF STABILIZING THERMO-PLASTIC CONTAINERS

This invention relates to an improved stabilized thermoplastic container and method of making the same.

All thermoplastics have a (thermal) memory point and when they are caused to extend beyond such (thermal) memory point they remain dimensionally stable only for an unknown and indeterminate length of time, dependent upon the temperature to which the material is subsequently subjected. This is particularly true when service temperatures (temperatures to which the bottle is subjected to during its useful life) exceed 110°F. or thereabouts.

It is known that stress patterns which are inherently set up during the process of manufacture, and which create thermally unstable thermoplastic products must be substantially removed, if service conditions demand subjection of a moulded thermoplastic container to temperatures at or above 110°F. In such processes, commonly referred to as annealing process, there is an alleviation of internal stress to a level which creates a new point of stability above 110°F., but substantially less than the environmental temperature to which the thermoplastic is subjected during annealing. It is for this particular reason that annealing processes are used, and why polyethylene bottles and other thermoplastic containers are subjected to heat treatment by conduction or convection over specific time bands; namely, to attempt to stabilize the container by alleviating the internal stresses created during manufacture. Hot air ovens are often used in annealing as well as water-boiling emersion and the like, controlled under a combination of temperature and related time during emersion.

It is known that thermoplastic containers or bottles, when manufactured by the process of blow moulding, vacuum forming or other present-day techniques, substantially reduce in capacity as the result of annealing. It is also known that stability of such annealed thermoplastic containers, is maintained at environmental conditions substantially less than the temperature employed during the annealing period, i.e. a bottle annealed by the convention method as taught by Bailey in his Canadian Pat. No. 787,822 issued 18 June 1968, entitled "Volume Stabilization of Moulded Plastic Containers" when subject to an annealing temperature of up to 200°F. for a time period of between 20 to 30 minutes has an empirical thermal stability level at or about 150°F. When such bottle is subject after annealing to temperatures at some point above the stability level of about 150°F. the bottle again is further fugitive in either or both dimension and capacity, i.e. retained stresses are further relieved within the bottle, causing a fugitive reduction in its capacity as well, in some instances, distortion of the container.

It has now been found that when thermoplastic bottles and the like are subject to washing for the purposes of re-use or which are to be filled with a product at about 150°F., the same must be stabilized to retain the desired capacity limits and requirements at the requisite temperatures in excess of 150°F. temperature. It has been found that thermoplastic containers, particularly of polyethylene, can be stabilized with the desired or required control limits to a controlled and determined thermal stability service level, that is to a

thermal stability which will sustain the high environment temperature provided the service temperature is less than the moulding temperature of the thermoplastic. This is accomplished by subjecting a thermoplastic container, preferably a thermoplastic which is not opaque to microwave radiation selected, to the action of microwave treatment for a substantially shorter period of time than required by any other process taught and currently practiced.

It has been further found that an expeditious method, to achieve the purpose of my invention, namely stability or integrity of thermoplastic containers, comprises the placing of a controlled amount of liquid such as water in the bottom of a thermoplastic container, such as a polyethylene bottle (the polyethylene acting essentially as a "window" to the microwave radiation, i.e. absorbs negligible microwave radiation) and the placing of the partially filled bottle within a microwave radiation field, for example, that generated by microwave ovens having output in the microwave broadcast range. The contented liquid (water) rapidly reaches its vaporization temperature, and is converted into steam due to the absorption of radiant energy by the liquid, while the polyethylene acts substantially as a "window" permitting the radiation to pass through the container wall to the liquid. As a result the liquid contained is elevated in temperature until it reaches beyond its boiling point and then vaporizes off. The elevated temperature of the liquid, and, in its gaseous phase, the vapor phase, scavenges the inner surface of the bottle heating the same to or above the boiling point of the liquid, in the instance of water above 212°F. when measured at sea level. The gaseous phase of the liquid also absorbs radiant energy and is further elevated in temperature (superheated) to further enhance the thermal scavenging of the inner surface of the bottle.

The invention therefore contemplates a method of stabilizing thermoplastic products beyond its initial or primary memory point (thermal deformation point) but below the plastic deformation point comprising the steps of:

a. selecting a thermoplastic product which is to be stabilized to a temperature beyond its primary memory point;
b. placing a liquid absorbant to microwave radiant energy in intimate proximity to the surface of the thermoplastic product;
c. subjecting the liquid to microwave radiant energy such that the liquid absorbs radiant energy and is elevated to a temperature in excess of the (first) primary thermal deformation point of the thermoplastic such that the thermo-plastic is also heated to a temperature above that which created the first memory point whereby the thermoplastic is re-established into a new primary (second) memory point above the first.

The invention will now be described by way of example reference being had to the accompanying drawings in which:

FIG. 1 is a schematic of a production method of utilizing the embodiments of the invention.

FIG. 2 is a three-part graph illustrating the net capacity in cubic centimeters of thermoplastic (polyethylene) containers prior to annealing and sub-

sequent to annealing wherein the annealing processes used are two of the processes of the prior art, a 3 hour boil — A, and a 30 minute conduction heat shrink method — C, and a third (B) the process embodying the invention.

FIG. 3A is a three part comparison graph illustrating the net capacity in grams of the containers of FIG. 2A after shrinking — post boil — and after one, five and 10 washings for five minutes at 168°F to 172°F.

FIG. 3B is a three-part comparison graph illustrating the net capacity in grams of the containers of FIG. 2B after shrinking — post microwave — and after one, five and 10 washings for five minutes at 168°F. to 172°F.

FIG. 3C is a three-part comparison graph illustrating the net capacity in grams of the containers of FIG. 2C after shrinking — post oven conduction — and after one, five and 10 washings for 5 minutes at 168°F to 172°F.

FIG. 4 is a comparative chart showing the total capacity lost in grams (averaged) for the containers of FIGS. 2A, 2B and 2C respectively during the 10 washings (after the initial annealing process).

FIG. 5 is a high-low total variation chart of the capacity losses in grams of the various containers.

It should be noted that in the above graphs, and the below described invention, a datum was selected as 3785 grams weight being equivalent to one United States of America gallon.

Particularly, in order to demonstrate the efficacy and the utility of the invention, it must be appreciated that thermoplastic bottles or containers which are commonly in existence in the dairy industry in North America are subject during their useful life span to temperatures in excess of 150°F. as a result of either dairy or consumer washing practices. It might be mentioned in passing that the North American dairy industry claims as a standard for dairy washing temperatures at between 140° to 150°F. It has been found that such practices are tempered by the economies of higher wash temperature and the demands of good sterile practices as may from time to time be prescribed by the appropriate governing bodies and agencies. As a result washing temperatures in dairies, generally exceed the 150°F. maximum, and usually range in the vicinity of 165° to 170°F.

As a result, if the memory point of thermoplastic diary bottles are retained in the neighborhood of 150°F., as they commonly are under the present state of the art, and such bottles are subjected during any washing cycle to temperatures in excess of 150°F. this initiates uncontrollable movement of the body material of the container construction causing an alteration in the volumetric capacity of the said bottle. Such alteration of the structural and capacity integrity continues after each washing or hot rinsing cycle, causing an unpredictable variance reflected in a new or re-established volumetric capacity after each wash.

Referring to FIG. 2 36 thermoplastic bottles having internal capacity slightly in excess of one United States of America gallon, were taken off the production line immediately after manufacture thereof. Such bottles were designed to contain an annealing allowance of approximately four ounces over the desired final capacity of one U.S. gallon (3785 grams), all bottles were nevertheless measured for true capacity of contents by weight. The bottles were marked, and the true capacity of each bottle was plotted respectively on the graphs shown in FIG. 2 (solid line). The bottles were then grouped into three equal groups (A, B, C) and the first group, A, was subjected to a normal heat treatment in a boiling water bath, as is often practiced in the art, for 3 hours at 205°F. The capacity of the contents, by weight, of these containers were once again taken and respectively plotted in FIG. 2A (dotted lines).

A third group of bottles was taken, group C, and the volumetric capacity, by weight, of these bottles was measured and plotted on FIG. 2C. These bottles were then subjected to heat stabilization by the conduction method as taught by Bailey in his Canadian Pat. No. 787,822 issued on 18 June, 1968, entitled Volume Stabilization of Moulded Plastic Containers. Such bottles were subjected for a period of thirty minutes to temperatures in the range of 198° to 205°F. After stabilization by the Bailey method the volumetric capacity, by weight, of the respective containers was again taken and the respective values plotted as shown in FIG. 2C by the dotted lines.

The second group B of bottles was likewise measured, for true capacity of contents, by weight, and the same plotted on FIG. 2B (solid lines) thereafter these bottles were stabilized according to the embodiments of the invention now to be disclosed, in the following manner. That is to say, 4 ounces of water were placed in each bottle and the charged bottle was then placed in a radio frequency microwave field, microwave radiant energy field, as generated by microwave ovens having output in the microwave broadcast range of 890; 940; 2,400; 2,500 M. H_z. or other approved microwave broadcast bands. After about 30 seconds the water in each bottle started to boil and steam could be seen coming out of the top. After about ten more seconds steam could not be seen emanating from the bottle but the level of the water in the bottle could be seen diminishing in volume. After approximately one minute more or less all the water had evaporated from within the bottle. After three minutes the radiation was turned off and the bottle removed from the oven. After cooling, the volume, by weight, of the bottle was again taken and respectively plotted on the graph of FIG. 2B (dotted lines).

Now referring to FIG. 2 it can be seen that bottles subjected to the three hour boiling, FIG. 2A, all save one, lost sufficient capacity on boiling that their net capacity was below datum (3785 grams) and in six instances less than 14 grams below datum (fourteen grams representing one-half fluid ounce). Referring to bottles subjected by shrinkage according to the embodiments of the invention it can be seen that only three bottles shrunk in capacity below datum and that the total deviation of upper limit (12 grams above datum) and lower limit (7 grams below datum) was less than the deviation with respect to those bottles subjected to the 3 hour boil, FIG. 2A. Bottles subjected to the Bailey heat conduction method of stabilization showed that four bottles stabilized to a volumetric capacity below datum, one in excess of the fourteen grams below datum. Further, one bottle shrunk to a capacity of 29 grams above datum, 28 grams representing one fluid ounce. It is to be noted at this time that in some countries, such as Canada, the governing authori-

ties do not permit certain containers to be on the market which have volumetric capacities less than one-half ounce below their respective datum, or in excess of 1 ounce of their respective datum.

It therefore can be seen, comparing the three techniques, that the prior art, of the 3 hour boil, FIG. 2A, and the heat conduction method of Bailey, FIG. 2C, generate bottles some of which are beyond acceptable limits. On the other hand bottles stabilized according to the invention all showed sufficient stabilization to be within the acceptable limits of datum.

After stabilization all bottles were subjected to ten washing cycles in duration of five minutes each at a controlled temperature of 170°F. (168°–172°F.). This wash did not contain any detergent or soap and might be considered as a hot rinse. Nevertheless, it was designed to simulate actual dairy washing cycle conditions as found in a dairy. After each wash the capacity of each bottle was measured, and respectively tabulated in FIG. 3 according to the method in which initial stabilization had occurred namely bottles subjected to the three hour boil method of stabilization were tabulated in FIG. 3A, bottles stabilized according to the embodiments of the invention were tabulated in FIG. 3B, and bottles stabilized according to the Bailey heat conduction process were tabulated in FIG. 3C. The tables only indicate the resultant capacity after one wash, five washes and 10 washes respectively. In any event careful examination of the results indicates that the capacity of bottles, irrespective of the process of stabilization, migrate to some extent during each wash. Comparing specifically bottles washed according to the embodiments of the invention, FIG. 3B with the bottles washed according to the prior art, FIGS. 3A and 3C, it can be seen that the migration of bottles washed according to the invention is less than migration according to the prior art. Specifically, after ten washings, bottles stabilized according to the 3 hour boil, FIG. 3A, all, save one, reached or exceeded the lower limit, 14 grams below datum, and as a result were not serviceable. Moreover after the first wash all but two bottles were beyond the lower limit of datum.

Referring to FIG. 3C and bottles stabilized according to the Bailey heat conduction method one bottle showed migration below the lower limit of datum after the first wash and it remained below that limit during all washes. All other bottles seemed to be satisfactory within the acceptable limits about datum. Referring to FIG. 3B after the first wash one bottle reached a capacity of the lower acceptable limit and it showed continual reduction in capacity beyond the lower acceptable limit after the first washing. All other bottles remained within acceptable limits during successive washes.

Now, referring to FIGS. 4 and 5 and particularly FIG. 5 is was seen that the hi-low total variation which bottles migrate during the washing cycle is less with bottles stabilized according to the embodiments of the invention than with either the two prior art techniques. In fact careful analysis shows that bottles shrunk by the Bailey heat conduction method shrunk 80 c.c. during the 30 minutes of stabilizing. After ten washes they shrunk an additional 7 c.c. approximately. On the other hand bottles shrunk according to the embodiments of the invention shrunk 106 c.c. during the 3 minutes of stabilization and only approximately 3 c.c. during the 10 washings. Nevertheless it must be admitted that the total aggregate capacity lost after 10 washings, with bottles stabilized according to the embodiments of the invention is somewhat higher than with bottles stabilized according to Bailey, see FIG. 4, it is nevertheless less than bottles stabilized according to the 3 hour boil method.

From the above results it is clear that thermoplastic bottles or containers stabilized according to the invention are significantly more stable and have a higher integrity than those of the prior art. They are thus capable of being used in environment temperatures in the range of 170°F. Such containers are therefore particularly useful for food and other products which are elevated in temperature above 150°F, in order to decrease their viscosity and to make then pourable, for example, jellies, jams and the like, but below the thermal distortion point of the thermoplastic. Furthermore, dairy bottles which are subjected to the stabilization processes embodying the invention will have a greater inherent stability and integrity to maintain volumetric capacity than bottles presently existing in the dairy industry.

Now referring to FIG. 1, thermoplastic bottles **10** which have just been moulded to an interior capacity slightly larger than the final capacity designed may be placed as by a hand **11**, on a continuous conveyor belt **12**. The bottle **10**, is then carried to beneath a nozzle **13**, which injects into the bottle **10**, a microwave absorbing fluid such as water **14**. The bottle **10**, then progressively moves into a microwave oven **16**, which emits radiant microwave energy **17**. The energy **17**, passes through the bottle **10**, essentially unabsorbed but is absorbed by the water **14**, elevating it in temperature to its boiling point. The steam (vapor) **18**, thereby generated is then superheated, scavenges the inside of the bottle **10**, and shrinks or stabilizes the bottle. At the same time the excess steam **18'** (vapor) emanates from the top of the bottle pervades the inner part of the oven. As a result this steam might have some scavenging effect on the outside surface of the bottle **10**, (but this is difficult to ascertain at the present). The bottle **10**, then is carried out of the oven **16**, all or some of the water having been evaporated, and the same is then removed from the conveyor belt **12**, as by hand **19** or automatically.

It will be appreciated that though the invention has been described in one embodiment utilizing a bottle of polyethylene thermoplastic, other thermoplastics can be used for the bottle. In such cases, the thermoplastic must have properties which permit it to act essentially as a "window" to the microwave radiant energy used. This allows the liquid to absorb the radiation and thereby to be elevated in temperature.

If, on the other hand, thermoplastics which are opaque to microwave radiation are used, i.e. thermoplastics which do not act as "windows," the use of a liquid absorbent to microwave radiation need not be as critical.

It has been found that for the preferred results that the polyethylene bottles, above described, are satisfactorily stabilized using water as the liquid. The amount of water to be used may vary with the size of the bottle to be stabilized; nevertheless, it would appear that for

bottles having a volume of a pint, a minimum amount of water to be used is about 1 ounce.

It might also be mentioned that a different selection of liquids with higher vapourization temperatures will be necessary as more sophisticated thermoplastics with higher plastic deformation points are used, if it is desired to increase the range of environmental and service temperatures for thermoplastic products.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method of dimensionally stabilizing a thermoplastic polymeric container as to capacity and shape by heating the container wherein the improvement comprises:

providing a container having an internal cavity and composed of a thermoplastic polymeric material which is generally translucent to at least one frequency of microwave radiant energy, said container having a capacity greater than the capacity to which it is to be stabilized;

introducing into said cavity a material consisting essentially of a liquid which is generally absorbent to such microwave energy to which said container is translucent, said liquid having a vaporization temperature greater than the primary memory temperature of the thermoplastic material and less than the plastic deformation temperature of the material,

selecting a microwave radiant energy field which has a frequency which is generally absorbed by said liquid and to which the thermoplastic material is generally translucent,

subjecting said container and said liquid to said field to elevate said liquid to its vaporization temperature and to vaporize said liquid, so as to heat said container to a temperature in close proximity to the vaporization temperature to establish a second memory temperature for the container above that of the first memory temperature.

2. The method of claim 1 wherein the liquid is water.

3. The method of claim 1 wherein the thermoplastic is polyethylene.

4. The method of claim 3 wherein the liquid is water.

5. The method of claim 3 wherein the microwave radiant energy field is selected from the group of microwave energy frequencies consisting of 890 megahertz.; 940 megahertz.; 2400 megahertz; and 2500 megahertz.

6. The method of claim 4 wherein the microwave radiant energy field is selected from the group of microwave energy frequencies consisting of 890 M. H_z.; 940 M. H_z.;, 2400 M. H_z.; and 2500 M. H_z.

7. A method of dimensionally stabilizing a polyethylene container as to capacity and shape by heating the container wherein the improvement comprises:

providing a container having an internal cavity and composed of polyethylene which is generally translucent to at least one frequency of microwave radiant energy, said container having a capacity greater than the capacity to which it is to be stabilized;

introducing into said cavity a material consisting essentially of water which is generally absorbent to such microwave energy to which said container is translucent, said water having a vaporization temperature greater than the primary memory temperature of the polyethylene and less than the plastic deformation temperature of the polyethylene;

said polyethylene having a thermal deformation temperature in excess of 180°F,;

selecting a microwave radiant energy field which has a frequency which is generally absorbed by said water and to which the polyethylene is generally translucent;

subjecting said container and said water to said field to elevate said water to its vaporization temperature and to vaporize said water so as to heat said container in close proximity to the vaporization temperature to establish a second memory temperature of approximately 160°F. for the container above that of the first memory temperature.

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