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(71) Applicant *(for all designated States except US)*: TETRA LAVAL HOLDINGS & FINANCE, S.A. [CH/CH]; 70, avenue Général–Guisan, CH–1009 Pully (CH).

(71)(72) Applicant and Inventor: FRISK, Peter [SE/US]; 2300 Lincoln Park West, Chicago, IL 60614 (US).

(74) Agent: CATANIA, Michael, A.; Tetra Pak–US, Intellectual Property Dept., 909 Asbury Drive, Buffalo Grove, IL 60089 (US).

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(54) Title: OXYGEN SCAVENGING CONTAINER

(57) Abstract

A container composed of a polymer material integrated with an oxygen scavenging agent is disclosed that is suitable for oxygen sensitive contents. The novel container is capable of scavenging excess oxygen from the enclosed atmosphere of the container without substantially modifying the design of similar container. The container includes at least one layer composed of a polymer material integrated with an oxygen scavenging agent between 0.01 and 1.0 % weight of the entire container. The oxygen scavenging layer only surrounds the atmosphere of the container while the rest of the container has an unmodified layer of the same polymer material. In most container configurations, the modified layer would be the neck portion of the container. The polymer material may be selected from polyethylene terephthalate, a copolymer of polyethylene terephthalate and a mixture thereof. The oxygen scavenging agent may be selected from iron base compounds, organic compounds and biologically active compounds. More specifically, the iron based compounds may be selected from pure iron, iron containing organic compounds, FeO_X, and $Fe_XO_Z(OH)_T$. The organic compounds used as oxygen scavenging agents may be selected from ascorbic acid, vitamin E, vitamin B and most other vitamins. The oxygen scavenging layer is in direct contact with the gaseous contents of the atmosphere of the container. The present invention also discloses a method for fabricating an oxygen scavenging container.

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TITLE

OXYGEN SCAVENGING CONTAINER

Technical Field

The present invention relates to a container composed of a polymer material integrated with an oxygen scavenger agent. Specifically, the present invention relates to a container composed of polyethylene terephthalate or a copolymer thereof, integrated with a oxygen scavenging agent in the upper portions of the container.

Background Art

In the packaging industry, the permeability of containers to oxygen has been the motivating factor for a number of inventions. Excess oxygen in a container for a food product will eventually lead to the degradation of the food product. For example, excess oxygen in a wine container will lead to the oxidation of the wine which will result in the formation of acetic acid, vinegar, thereby destroying the value of the intended food product, wine. Other oxidation reactions are equally destructive to a plethora of food products which provides the motivation for those in the industry to invent different methods to overcome the problem with oxygen permeability. One method has been to prevent the ingress of oxygen into the packaging by creating packaging materials with enhanced impermeability which substantially, but not entirely, prevent the ingress of oxygen into the container. Another method has been to remove the oxygen once it has entered the container through use of an oxygen scavenger.

Various techniques have been developed to scavenge oxygen from containers using an assortment of scavenging agents. One such technique is to place the oxygen scavenging agent into one layer of the packaging material, then cover this scavenging layer with a oxygen permeable layer thereby preventing contact between the scavenging layer and the contents while allowing for the removal of oxygen from the container. Farrell et al, U.S. Patent No. 4,536,409, for an Oxygen Scavenger, discloses such a technique. In Farrell et al, a polymeric layer containing the oxygen scavenger agent is matched with a permeable protective layer thereby permitting removal of the oxygen without having any direct contact between the contents and the oxygen scavenging layer. Speer et al, U.S. Patent No. 5,350,622, for a Multilayer Structure For A Package For Scavenging Oxygen also discloses a container for food which includes a barrier layer, a oxygen scavenging layer, and an innermost permeable layer which prevents contact between the contents and the oxygen scavenger. Although these inventions have the ability to scavenge oxygen from a container, they also increase the number of layers for the container to prevent contact between the scavenging agent and the contents.

Most containers for food products are not completely filled, thereby creating a space for the gaseous contents to reside when the container is sealed. Due to its partial pressure, oxygen prefers the gaseous state and will migrate from the solid or liquid phase contents to this space inadvertently created for the gaseous contents. In a bottle, this space would encompass the neck of the bottle and the space immediately below the neck. Therefore, the oxygen scavenging agent should also be located in the neck of the bottle since the majority of the excess oxygen will reside in this space.

Several inventions have come forth which attempt to take advantage of oxygen's preference for the gaseous state. Schvester, U.S. Patent No. 4,840,280, for a Sealing Cap For Liquid Food Or Beverage Containers discloses a sealing cap for a container for a liquid contents having a sealed bag containing the scavenging agent wherein in the sealed bag is placed within the permeable layers of the cap. In this manner, Schvester attempts to scavenge oxygen from a container. Morita et al, U.S. Patent No. 4,756,436, for a Oxygen Scavenger Container Used For Cap also discloses a cap for a container for a liquid contents which has an oxygen scavenger placed within a number of permeable layers. These caps, similar to the above-mentioned packaging materials, disclose a cap composed of a multitude of layers which increase the size and costs of the caps, and also add to the complexity of the fabrication process.

The foregoing patents, although efficacious in the scavenging of oxygen, are not the denouement of the problems of excess oxygen in containers. There are still unresolved problems which compel the enlargement of inventions in the scavenging of excess oxygen from containers.

Disclosure of Invention

The present invention enlarges the scope of scavenging excess oxygen from containers by providing an approach to this problem which does not increase the number of layers of a container, nor does it increase the complexity of the fabrication process. The present invention is able to accomplish this by providing a novel container composed of a polymeric material wherein only the polymeric material of the container encompassing the gaseous contents is integrated with an oxygen scavenging agent.

One embodiment of the present invention is a container capable of scavenging excess oxygen from an atmosphere of the container. The container comprises at least one layer substantially surrounding the atmosphere of the container. The at least one layer is composed of a polymer material integrated with an oxygen scavenging agent between 0.01% and 1.0 % weight of the container. The polymer material may be selected from the group consisting of polyethylene terephthalate, a copolymer of polyethylene terephthalate, and a mixture thereof. The oxygen scavenging agent may be selected from the group consisting of an iron based compound, an organic compound, and a biologically active compound. The iron based compound may be selected from the group consisting of FeO_X, pure iron, iron containing organic compounds and $Fe_XO_Z(OH)_T$. The oxygen scavenging agent may be activated by exposure to a relatively high humidity environment. The atmosphere of the container may be composed of a gaseous contents which are predominantly water vapor, nitrogen, carbon dioxide and oxygen. The container may also be composed of a multitude of layers. The organic compound may be selected from the group consisting of ascorbic acid, vitamin B and vitamin E.

Brief Description of Drawings

There is illustrated in FIG. 1 a cross-section view of one embodiment of a container of the present invention;

There is illustrated in FIG. 2 a cross-section view of an alternative embodiment of a container of the present invention;

There is illustrated in FIG. 3 a cross-sectional view of one embodiment of the multiple layers of a container of the present invention.

Modes for Carrying Out the Invention and Industrial Applicability

Containers for flowable food products such as fruit juices, alcoholic beverages, soups and the like usually provide for an "atmosphere" in the sealed container. This atmosphere, which is composed of gaseous contents, usually lies above the primary contents of the container and serves several purposes. One purpose may be to reduce the amount of the primary contents of the container as a costs saving measure to the manufacturer. Another purpose may be to serve as a safety measure to accommodate variations in pressure the container may undergo during distribution. Still another purpose may be to provide the consumer with a container which will not spill its contents during the opening of the container. Although this atmosphere may serve many purposes, it may also present problems for the manufacturers. One such problem pertains to excess oxygen in the container. Excess in that the oxygen is not needed by the contents of the container and in fact is most likely detrimental to the contents of the contain.

The container of the present invention is designed to remove the excess oxygen from the atmosphere of the container in a novel manner which does not greatly increase the costs or complexity of fabricating containers for flowable food products.

The container of the present invention is composed of a modified polymeric material which is capable of scavenging excess oxygen from the atmosphere of the container. The modified polymeric material is PET, COPET or a mixture thereof integrated with an oxygen scavenging agent. The oxygen scavenging agent is integrated with PET, COPET or a mixture thereof before the modified polymeric material is converted into a container configuration such as a bottle. One of the novel aspects of the present invention is the minimal amount of an oxygen scavenging agent necessary to effectively remove excess oxygen from the atmosphere of the container. The present invention only requires a minimal amount of oxygen scavenging agent since only the upper portion of the container which surrounds the atmosphere is actually composed of the modified polymeric material while the rest of the container is composed of an unmodified polymeric material. This upper portion of the container, sometimes referred to as the "headspace," is where oxygen prefers to reside in the container due to the partial pressure of oxygen. Therefore, by taking advantage of oxygen's preference for the gaseous state, the present invention only requires a minimal amount of oxygen scavenging agent to effectively prevent the oxidation of the primary contents of the container.

The oxygen scavenging agent is integrated with the polymeric material in an amount of approximately 0.01 to 1.0 weight percent of the entire container. The oxygen scavenging material may be selected from one or more materials including: an organic compound; an iron-based compound; and/or a biologically active compound. The iron- based compound may include FeO_x, pure iron, an iron containing organic compound and $Fe_xO_y(OH)_z$. The use of iron-based compounds allow the oxygen scavenging agent to be humidity activated at a time prior to or concurrent with the filling of the container. For example, subsequent to the fabrication of the container, the container may be stored indefinitely in a relatively low humidity environment. Then, prior to or concurrent with the filling process, the container may be exposed to a relatively high humidity environment for a predetermined time period sufficient for the activation of the oxygen scavenging agent. A further, iron based oxygen scavenging compound suitable for use in the present invention is OXYGUARD which is available from Toyo Seikan Kaisha of Yokahama, Japan.

Various organic compounds which are well known by those skilled in the pertinent art may be utilized as oxygen scavenging agents for the present invention. For example, ground sea grass and/or ground tea leaves may be suitable for use as an oxygen scavenging agent for the present invention. Also, a rice extract, such as disclosed in Tokuyama et al, U.S. Patent No. 5,346,697, for an Active Oxygen Scavenger, may be utilized as an oxygen scavenging agent for the present invention. Further, most vitamins may be used as oxygen scavenging agents in practicing the present invention. Specifically, an ascorbic acid (vitamin C), a vitamin B or a vitamin E compound may be used as oxygen scavenging agents in practicing the present invention.

Monomers and short chain polymers of, for example, polypropylene and/or polyethylene are likewise organic compounds which are suitable as oxygen scavenging agents for utilization in practicing the present invention. If a short chain polymer is utilized, selective activation of the oxygen scavenger agent is possible by irradiating the modified polymeric material with, for example, ultraviolet light or with electron beam emissions. Such irradiation effects a cutting of the inter-monomer bonds thereby creating even shorted, and more chemically active, polymer chains and monomers. If acceleration of the oxygen scavenging process is desirable, a mixture of both organic compounds and iron-based compounds may be integrated into the polymeric material which in a preferred embodiment is either PET, COPET or a mixture thereof.

Once the modified polymer material is formed, the oxygen scavenging container may be fabricated through a number of molding methods. Although the novel oxygen scavenging container of the present invention has the capability to remove excess oxygen from the gaseous contents of the container, the novel container may be fabricated in a similar fashion to containers fabricated from unmodified PET or COPET resin with only minor adjustments to the molding processes.

Three methods for manufacturing containers from PET or COPET resin are extrusion molding, injection molding and thermoforming. One extrusion method is extrusion blow molding wherein the parison is extruded and blow molded to the final bottle configuration. Another method is extrusion stretch blow molding wherein the parison is extruded and cooled to a wall temperature range of approximately 90-125 °C, then blow molded to the final bottle configuration. Still another method is two stage extrusion stretch blow molding wherein the parison is first extruded and cooled to room temperature. Then, the parison is transported to a separate operation where it is reheated to a wall temperature of 90-125 °C and then blow molded to the final bottle configuration.

An injection method is injection blow molding wherein a parison is injected molded and then the hot parison is blow molded to the final container configuration. Yet another injection method is injection stretch blow molding wherein a parison is injection molded and cooled to a wall temperature of 90-125 °C before being stretch blow molded to the final container configuration. A final method is two stage injection stretch blow molding wherein a parison is injection molded and cooled to room temperature. Then, transported to a separate operation where it is reheated to a wall temperature of 90-125 °C and then stretch blow molded to the final container configuration.

Thermoforming is a low pressure process that converts flat, basically two-dimensional thermoplastic sheet stock into larger, generally more complex three dimensional containers. The thermoforming process begins with sheets that are cut to size, then loaded and clamped into a thermoforming machine. The sheet is heated to a softening temperature and formed into a container. The containers are cooled, unloaded from the machine and trimmed to remove any extra material.

A preferred method of fabricating the oxygen scavenging container is through two-stage injection stretch blow molding, however any of the previously mentioned molding processes will suffice to fabricated an oxygen scavenging container embodied in the present invention.

As shown in FIGS. 1 and 2, a container is generally designated 10. Although the container 10 is in the shape of a bottle, such shape is for illustration purposes and is not intended to limit the possible configurations for the present invention. The container 10 consists of a lower portion 12 and an upper portion 14. The container 10 also has an opening 16 located at the top of the container 10.

The lower portion 12 generally encompasses the area filled by a primary contents 18 of the container 10 which is the area beneath dashed lines 19 which represent the normal fill level of such a contianer 10. The primary contents 18 may be a liquid such as a carbonated beverage, water, fruit juice and the like. The primary contents 18 may also be a solid such as a granular spice. Further, the primary contents may be a combination of a liquid and a solid such as a soup or yogurt. The lower portion 12 is composed of a polymer material which is substantially unreactive with the primary contents 18 of the container 10. In a preferred embodiment , the lower portion 12 is composed of PET, COPET or some mixture thereof. However, alternative embodiments may have a modified PET, COPET or mixture thereof which enhances the inherent properties of such materials.

The upper portion 14 generally encompasses a gaseous contents 20 of the container 10. In the bottle configuration illustrated in FIG. 1, the upper portion 14 is the neck portion of the bottle. The gaseous contents 20 will most likely be gases entrapped in the container 10 after sealing of the opening 16 and gases permeating from the primary contents 18. The gaseous contents 20 may also be gases which permeated through the container 10 from either the lower portion 12 or the upper portion 14. The gaseous contents 20 will predominantly include oxygen, carbon dioxide and water vapor. The upper portion 14 is composed of a modified polymer material which is capable of a scavenging oxygen from the gaseous contents 20 thereby reducing the possibility that the oxygen will adversely react with the primary contents 18. The modified polymer material has an integrated oxygen scavenging agent which binds with any excess oxygen thereby removing it from the gaseous contents 20. The polymer material is PET, COPET or any mixture thereof, and the oxygen scavenging agent is integrated into the polymer material during the polymer fabrication, or during the film production stage.

In a preferred embodiment, the upper portion 14 is composed of one layer of the modified polymer material which is in direct physical contact with gaseous contents 20. However, alternative embodiments may have a multitude of layers, and may have a layer which is an oxygen barrier layer. The upper portion 14 is located above the primary contents 18 to minimize the contact between the primary contents 18 and the oxygen scavenging agent integrated into the polymer material of the upper portion 14. Thus, the size of the upper portion 14 and lower portion 12 will be dependent on the size and shape of the container 10, and the level to which the primary contents 18 is filled within the container 10.

As mentioned previously, the container 10 may have a multitude of layers in addition to the layer of modified polymer material and unmodified polymer material. These additional layers may have enhanced barrier properties to prevent the ingress and egress of various gases including oxygen. As shown in FIG. 2, an additional exterior layer 22 surrounds the layer which is upper portion 14 and lower portion 12.

Alternatively, the container may have an interior layer to act as a barrier layer. As shown in FIG. 3, the cross-section of the container 10a has polymeric material layer 23 and a metal oxide deposition 24, with the deposition on the interior of the container 10a. The polymeric material layer 23 may have a thickness of approximately 25 to 150 microns. The polymeric material layer 23 is as described above with the oxygen scavenging modified polymeric material in the upper portion 14 and the unmodified material in the lower portion 12. The metal oxide deposition 24 may have a thickness range of approximately 5 to 500 nanometers. The stoichiometry of the metal oxide is important to maintain the transparency and the high barrier properties of the container 10a. In the formula MOx where M is either aluminum, silicon or iron, and x is between 1.8 and 2.5, the ability to deposit the metal oxide within this stoichiometric range prevents the container from becoming tinged thereby loosing its transparency. For example, when M is silicon and x is near 1.0, the container will have a yellow tinge indicative of silicon oxide, a semiconductor which has a relatively narrow electron band gap between a filled valence band and an empty conduction band thereby allowing for the absorption of light. Whereas when M is silicon and x is near 2, the metal deposition is silicon dioxide, an insulator which has a relatively large electron band gap between a filled valence band and an empty conduction band thereby allowing for the transmission of light. Thus, it is very important that the deposition of the metal oxide be performed in a manner that will ensure this stoichiometric range in order to have the transparency as well as the expected barrier properties.

The metal oxide deposition 24 may be deposited on the polymeric material layer 23 through a number of deposition methods. These methods include plasma-enhanced chemical vapor deposition, metalorganic chemical vapor deposition, halide transport chemical vapor deposition, liquid atmospheric photo chemical deposition, electron beam evaporation, pulsed laser ablation, atomic layer epitaxy, ion implantation, molecular beam epitaxy and RF magnetron sputtering. A preferred deposition method is plasma enhanced chemical vapor deposition described in Fayet *et al*, U.S. Patent No. 5,531,060 which is hereby incorporated by reference. However, it will be apparent to those skilled in the pertinent art that other deposition methods may be employed while not departing from the scope of the present invention.

One embodiment of the container 10 may have the polymeric material layer 23 composed of a biaxially oriented PET integrated with an oxygen scavenger, and a silicon oxide deposition 24 having the following stoichiometry, SiOx, where x has value between 1.5 and 2.5.

A method of integration occurs before and during polymerization of the polymeric material. The oxygen scavenger agents are added to the precursors materials (terephthalic acid and ethylene glycol) before the polymerization process to form a modified precursor material. Preferably, the oxygen scavenger agents should not affect the transparency of the PET. The modified precursor materials are reacted to form the modified pre-PET monomer. Using the TA process, this step is a direct esterification reaction.

An alternative pathway to obtain the modified pre-PET monomer has the monomer solution prepared through a direct esterification reaction using non-modified precursor materials. Then, the oxygen scavenger agent is dissolved into the monomer solution, before the polymerization process, to form the modified pre-PET monomer. Next, the modified pre-PET monomer is polymerized to form the integrated PET resin. Next, the integrated PET resin is converted to a neck configuration for a container. Next, unmodified PET resin is added to the neck configuration to complete the final container configuration. Although this example pertains to a neck configuration for a bottle, those skilled in the pertinent art will recognize that other configurations to encompass the gaseous contents of a container are applicable to other container shapes. The molding of the container may take place through many processes, including the above-mentioned molding processes.

A second method of integration occurs before or during the conversion of the PET resin into the final package design. The second method of integration may begin through two different pathways. First, the precursor materials for the TA process are reacted to form the monomer solution. Alternatively, the precursor materials for the DMT process undergo the first esterification reaction. Next, polymerization occurs to form a PET resin. Next, the oxygen scavenger agent is blended into the PET resin which results in a PET compound with a high concentration of the oxygen scavenger agent. The blending of the oxygen scavenger agent may be performed through use of a twin screw extruder. Next, the high concentration compound is then added to a larger amount of unmodified PET resin before conversion to the final package. Next, the PET resin, including the high concentration compound, is converted to the neck configuration. The conversion of the PET resin to the neck configuration also assists in blending and dispersing the high concentration compound throughout the PET resin, and ultimately the neck of the bottle. Next, unmodified PET resin is added to the neck configuration to complete the final container configuration. The molding of the container may take place through many processes, including the above-mentioned molding processes.

CLAIMS

I claim as my invention:

1. A container capable of scavenging excess oxygen from an atmosphere of the container, the container comprising:

at least one layer substantially surrounding the atmosphere of the container, the at least one layer composed of a polymer material integrated with an oxygen scavenging agent, the agent being between 0.01% and 1.0 % weight of the container, the oxygen scavenging agent selected from the group consisting of an iron based compound, an organic compound, and a biologically active compound and the polymer material selected from the group consisting of polyethylene terephthalate, a copolymer of polyethylene terephthalate, and a mixture thereof.

2. The container according to claim 1 wherein the polymer material has a metal oxide deposition on the exterior film having a thickness range of approximately 5 to 500 nanometers and having a formula of MO_X where x has a range of approximately 1.5 to approximately 2.5 and M is selected from the group consisting of silicon, aluminum and iron.

3. The container according to claim 1 further comprising a barrier layer juxtaposed to the at least one layer.

4. The container according to claim 1 wherein the iron based compound is selected from the group consisting of FeO_X, pure iron, $Fe_XO_Z(OH)_T$ and iron containing organic compounds.

5. The container according to claim 1 wherein the oxygen scavenging agent is activated by exposure to a relatively high humidity environment.

6. The container according to claim 1 wherein the organic compound is selected from the group consisting of ascorbic acid, vitamin E and vitamin B.

7. The container according to claim 1 wherein the at least one layer is in direct contact with the gaseous contents of the atmosphere of the container.

8. The container according to claim 1 wherein the at least one layer substantially prevents the degradation of a primary contents of the container.

9. The container according to claim 9 wherein the primary contents of the container are a liquid food product.

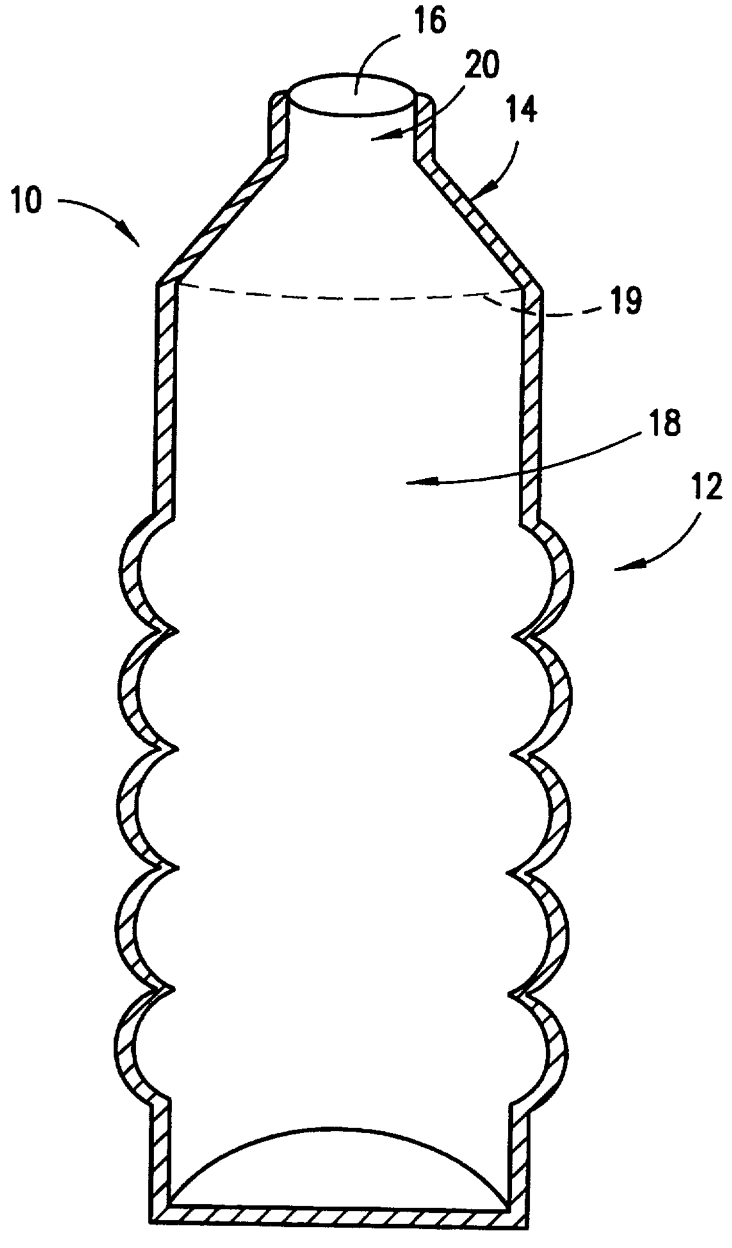

FIG. 1

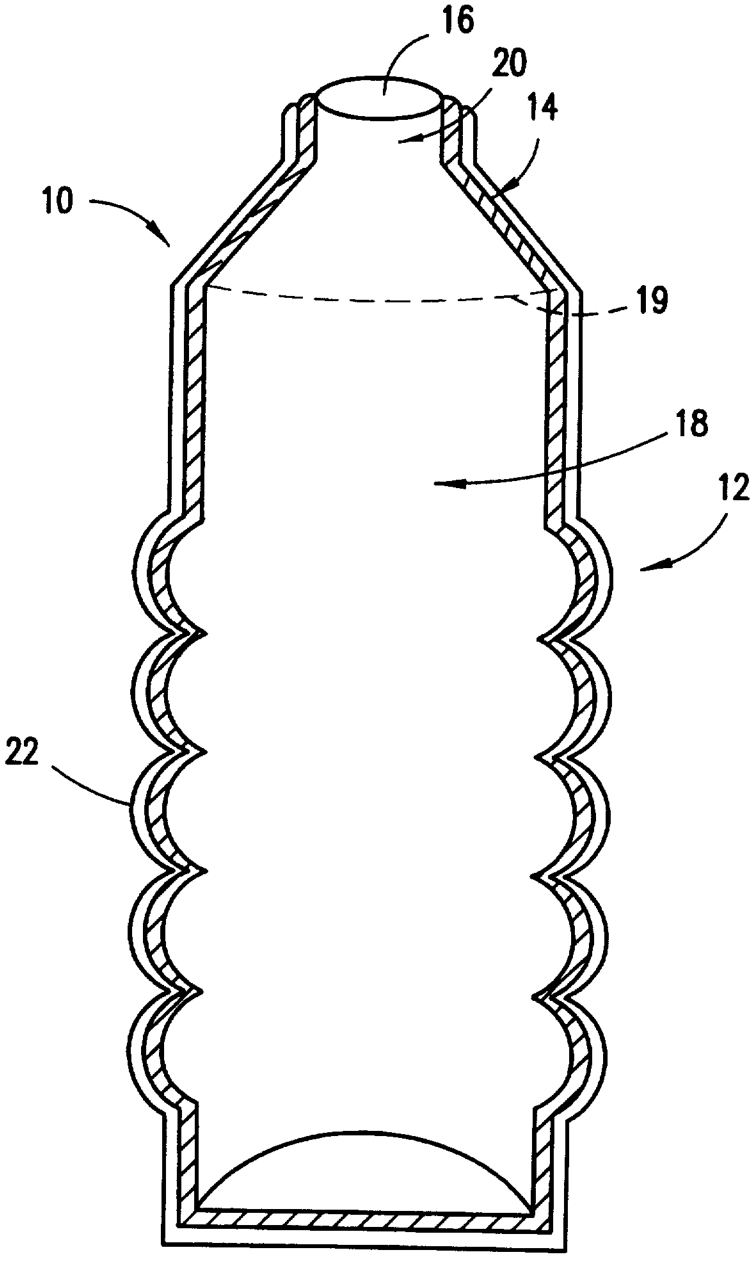

FIG. 2

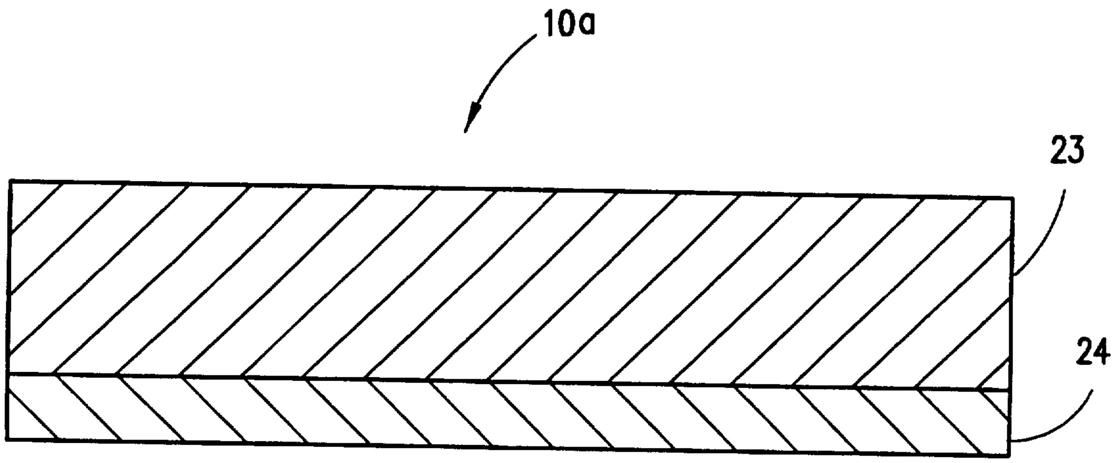

FIG. 3