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GASIFIED CONFECTION AND METHOD OF MAKING THE SAME

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This invention relates to a technique for enclosing a gas within a solid matrix and to the gas-containing solid so prepared.

The most common techniques for storing gases involve the use of containers wherein the gases are maintained under pressure. As is well known to those skilled in the art, effective use of this technique requires high pressures and accordingly strong containers. Typically, for example, gas containers are made of steel, and they may have a pressure therein of anywhere from e.g. 700 to 2000 p.s.i. Alternatively, it is possible to store gases by liquification or solidification, but these techniques are unsatisfactory in that they require low temperature, typically with accompanying high pressure.

It is an object of this invention to provide a technique for enclosing gas within a solid matrix. Other objects will be apparent to those skilled in the art on inspection of the following description.

According to certain aspects of this invention, it is possible to prepare a gas-containing solid which is substantially stable at room temperature for extended periods of time comprising a solidified fusible sugar containing therewithin a gas. Under preferred conditions, this product may be obtained by gassing a fused fusible sugar and cooling the said gasified fusible sugar below its fusion temperature.

According to certain preferred aspects of this invention, a gas may be enclosed within a solid matrix by the process which comprises fusing a fusible sugar, contacting said fusible sugar with gas at a pressure of 50–1000 p.s.i.g. for time sufficient to permit adsorption in said sugar of 0.5–15 ml. of gas per gram of sugar, maintaining the temperature of said sugar during said adsorption above the solidification point of said fused sugar, and cooling said sugar under pressure to a temperature less than its fusing temperature thereby obtaining a gas-containing solid.

The fusible sugars which may be employed in carrying out the process of this invention will preferably be those which fuse under the condition of operation at a temperature at least slightly above room temperature, so that they may readily be fused or melted to form a liquid which may readily be converted to a solid on return to and maintenance at room temperature.

Although the terms "fuse" or "melt" will be herein employed, it will be apparent to those skilled in the art that the fusion or melting point need not be a fixed precise point at which the material fuses or melts, but rather that these terms embrace a range or even a situation where the materials have no melting point at all in the strict physicochemical sense. The terms include the passage from a solid state to a liquid state and also the formation of a solution-liquid phase by the dissolving of solid in any water which may be present.

Included among the preferred fusible sugars are those which, on cooling, pass from the liquid phase to the solid phase by supercooling. Although we do not wish to be limited by this mode of description, it does appear that some of the preferred sugars exist in solid form as supercooled liquids with no sharp melting point.

It will be apparent that those fusible sugars which may be employed have a melting or fusion point below their

decomposition temperature, and that no substantial decomposition occurs at the melting or fusion temperature which would interfere with fusion, melting, or solidification on cooling.

Although as will be apparent from this disclosure, the fusible sugars which may be used in the practice of this invention include those which have a melting or fusing point which falls within a wide range, the preferred materials will be those having a melting or fusing point within the range of 25° C. to 150° C. or 200° C.

The fusible sugars which may be employed in the practice of this invention include sugars and their derivatives such as sugar alcohols and sugar acids. Typical fusible monosaccharide sugars include glucose, fructose, arabinose, etc. Typical fusible disaccharide sugars include sucrose, lactose, maltose, fructosan, etc. Typical fusible polysaccharide sugars include gentiobiose, cellobiose, panose, malto-triose, malto-tetrose, etc. Typical sugar alcohols include sorbitol and mannitol. Typical sugar acids include gluconic acid and saccharic acid.

The fusible sugars useful in connection with this invention will preferably be in near-anhydrous state. Although, for example, anhydrous sucrose having a melting point of 186° C., may be employed, it is found that the desired results may be readily obtained if a small percentage of water be present. If 1% water be present, the apparent melting or fusing point will be about 127° C.; if 2% water be present, the apparent melting or fusing point will be about 118° C. Typically the quantity of water present may be about 1%–5% and fusible sugars containing these amounts of water may be said to be substantially anhydrous.

Although individual sugars such as sucrose may be used, it is a feature of this invention that combinations of sugars, e.g. sucrose and lactose give preferred products. A mixture of 30% lactose with 70% sucrose gives an excellent carbonated, hard product particularly characterized by its low hygroscopicity and reduced stickiness on standing. A mixture of sucrose with corn syrup (containing glucose, maltose, dextrin) is also satisfactory.

It is also a feature of this invention that more gas may be retained in the solid product when it comprises a fusible mixture of sugar together with (*a*) dextrin; or (*b*) starch; or (*c*) gelatin; or (*d*) a gum, typified by agar agar, carragheenin, alginates, and pectin. For example, sucrose and dextrin may be employed together. In all cases the desired mixture is fusible as heretofore defined.

When the product is prepared by pressurizing a fusible sugar, it possesses a bright transparent appearance; it has a gas content of e.g. carbon dioxide of 0.5 to 4.5–5.0 ml. per gram; it is fast dissolving in water; it is heavier than water; and the bubbles liberated therefrom are quite large in size.

When the product is prepared by pressurizing a fusible sugar in combination with the other materials heretofore noted, it possesses an opaque or translucent appearance; it has a gas content of e.g. carbon dioxide of 5–15 ml. per gram; it is slow dissolving in water; it is lighter than water; and the bubbles liberated therefrom are initially large but subsequently much smaller in size. This product appears to be one wherein the gas may be more tightly bound in molecular encapsulation.

Other materials which may be added to the fusible sugar particularly when the product is to be used for edible purposes include: edible acids, typically citric acid, tartaric acid, adipic acid, lactic acid, fumaric acid, etc. (in quantity sufficiently small to eliminate or minimize inversion); buffer salts, typically citrates, tartrates, etc.; flavors, typically cherry, lime, cola, root beer, etc.; or coloring, typically red, brown, yellow, etc.

In carrying out the process of this invention, the gas-containing product may be prepared either continuously or batchwise. Under preferred conditions the fusible sugar and the other ingredients including e.g. desired acids, buffers, flavors, colors, etc., may be placed within an apparatus which is capable of withstanding the various temperatures and pressures prevailing during the course of the process. The temperature of the fusible sugar is then raised so that the sugar fuses.

The desired gas, which may be nitrogen or air, but which preferably is carbon dioxide, may be admitted to the system. Other gases such as ethylene oxide or inert gases including helium may be employed. When carbon dioxide is employed, it may be possible to add it in solid or liquid form; under preferred conditions, it will be admitted in gaseous form.

The incorporation of the gas into the fusible sugar under fusion-producing conditions may be effected by various techniques: extrusion, followed by hardening and release of pressure; molding under pressure; and various agglomerating techniques where fusion is effected by pressure; preferably it will be effected by mixing the gas with agitated fused sugar.

When the reaction is conducted batchwise and the carbon dioxide is to be added in gaseous form, the fusible sugar at temperature above its fusing point, is agitated and the carbon dioxide gas under desired pressure is admitted to the reaction chamber. Although the pressure of the gas may be varied somewhat depending upon prevailing conditions, it is preferred to maintain a superatmospheric pressure, i.e., a pressure of at least 50 p.s.i.g. and less than 1000 p.s.i.g. Preferred pressure is 400 p.s.i.g. to 800 p.s.i.g.

The time of contact of the liquid and the gas and the other conditions noted may vary somewhat depending on the particular characteristics of the system in which the reaction is carried out. Typically, however, the time of reaction will be controlled to give the desired amount of gas in the product—varying from e.g. 0.5–2.5 to about 15 ml. per gram of product. Typically the time of reaction will be of the order of 2–6 minutes, say 3.5 minutes.

At the end of the desired reaction time, the reaction mixture may be cooled under pressure to a temperature below that of the fusion temperature of the mixture. Preferably this will be done rapidly i.e. sufficiently quickly to minimize the crystallization of the sugar. It is found that rapid cooling increases the fragility, minimizes inversion, and reduces hygroscopic tendencies of the product.

The product of this invention may be stored under low relative humidity at temperatures which may approach e.g. 100° F. for extended periods of time e.g. one year. Despite the high level of gas within the product, and the fact that individual pieces may give off a loud noise as they explode under impact, the material may be stored without danger of explosion.

The product of this invention finds a wide variety of uses. It may be used for gas-storage uses where it is desired to store e.g. carbon doxide at room temperature at atmospheric pressure for extended periods of time. It may be employed as a carbonating agent in beverages, as a leavening agent in baking, etc. Other uses will be apparent to those skilled in the art.

It is a feature of this invention that the gasified product may be used as a "carbonated hard candy." In appearance it typically resembles hard sugar type candies. However, when the product of this invention is placed in the mouth, it disintegrates with a mild popping sound and liberates gas on contact with liquid and under pressure as it dissolves. This candy possesses a mouth feel which is totally unlike that which characterizes other products.

Example I

70 parts by weight of sucrose were mixed with 30 parts (dry base) by weight of 42 D.E. corn syrup. The liquid mixture was cooked at 160° C., the resulting mixture having a moisture content of 2%. This mixture was placed within a Parr reactor (a thick-shelled pressure vessel hav-

ing a stirrer) wherein it was maintained in fused condition at temperature above 100° C. Carbon dioxide gas to 600 p.s.i.g. was admitted and the mixture agitated for six minutes.

The reactor was rapidly cooled to 25° C. and opened. The product contained therein was hard and friable. It was found to contain 4.5 ml. of carbon dioxide per gram of product.

Example II

210 parts by weight of sucrose, 90 parts by weight of lactose, 60 parts by weight of water were placed within a pressurized reactor. The liquid mixture was cooked to 160° C. with stirring. The fused material having a moisture content of about 1%–2% or less was placed within a Parr reactor. Carbon dioxide at 600 p.s.i.g. was introduced and the mixture agitated for six minutes.

The reactor was rapidly cooled to room temperature, the pressure released, and the solid removed. The product had a carbon dioxide content of 4.5 ml. per gram of product. It was less hygroscopic than the product of Example I.

Example III

300 parts by weight of crystalline sorbitol were heated to 110° C. at which point it was fused. The fused material having a moisture content of about 1%–2% or less was placed within a Parr reactor. Carbon dioxide gas was admitted to a pressure of 600 p.s.i.g. and the fused sugar was agitated for 6 minutes in the presence of the gas.

The reactor was cooled rapidly to 25° C. and opened. The carbonated product contained therein was found to be glassy, hard, and friable, and to contain 4 ml. carbon dioxide per gram of product.

The product was heavier than water, it dissolved in water rather quickly and it had a characteristic snap and pop. The bubbles which were liberated on dissolution in water were found to be quite large in size.

Example IV

300 parts by weight of glucose were mixed with 60 parts by weight of water. The mixture was heated to 150° C. at which point it was fused. The fused material having a moisture content of 2% was placed within a Parr reactor. Carbon dioxide gas was admitted to a pressure of 600 p.s.i.g. and the fused sugar was agitated for 6 minutes in the presence of the gas.

The reactor was cooled rapidly to 25° C. and opened. The carbonated product contained therein was found to be hard and friable and to contain 4 ml. carbon dioxide per gram of product.

The product was heavier than water, it dissolved in water rather quickly and it had a characteristic snap and pop. The bubbles which were liberated on dissolution in water were found to be quite large in size.

Example V

69 parts by weight of sucrose were mixed with 30 parts by weight of 42 D.E. corn syrup, 41 parts by weight of agar agar, 40 parts by weight of water. The mixture was heated to 160° C. at which point it was fused. The fused material having a moisture content of 2–3% was placed within a Parr reactor. Carbon dioxide gas was admitted to a pressure of 600 p.s.i.g. and the fused sugar was agitated for 6 minutes in the presence of the gas.

The reactor was cooled rapidly to 25° C. and opened. The carbonated product contained therein was found to be hard and friable and to contain 12 ml. carbon dioxide per gram of product.

The product was lighter than water, it dissolved in water rather slowly, and it had a characteristic snap and pop. The bubbles which were liberated on dissolution in water were found to be initially large, but subsequently smaller in size.

Example VI

70 parts by weight of sucrose were mixed with 30 parts by weight of 42 D.E. corn syrup and 12 parts by weight of water. The mixture was heated to 160° C. at which point it was fused. The fused material having a moisture content of 2–3% was placed within a Parr reactor. Carbon dioxide gas was admitted to a pressure of 400 p.s.i.g. and the fused sugar was agitated for 6 minutes in the presence of the gas.

The reactor was cooled rapidly to 25° C. and opened. The carbonated product contained therein was found to be hard and friable and to contain 2.8 ml. carbon dioxide per gram of product.

The product was heavier than water, it dissolved in water rather quickly, and it had a characteristic snap and pop. The bubbles which were liberated on dissolution in water were found to be quite large in size.

Example VII

210 parts by weight of sucrose, 90 parts of 42 D.E. corn syrup, 40 parts of water, 0.2 part of cherry flavor and 0.1 part of strawberry color were mixed together. The mixture was heated to 160° C. at which point it was fused. The fused material having a moisture content of 2–3% was placed within a Parr reactor. Carbon dioxide gas was admitted to a pressure of 400 p.s.i.g. and the fused sugar was agitated for 6 minutes in the presence of the gas.

The reactor was cooled rapidly to 25° C. and opened. The carbonated product contained therein was found to be hard and friable and to contain 2.8 ml. carbon dioxide per gram of product.

The product was heavier than water, it dissolved in water rather quickly, and it had a characteristic snap and pop. The bubbles which were liberated on dissolution in water were found to be quite large in size. When this candy was placed in the mouth, it had a very unusual mouth feel as the gas was liberated. A tingling sensation was experienced and the overall effect was quite unlike that of any other candy or confection. As the candy dissolved, it gave a mild popping sound.

It will be apparent to those skilled in the art that various modifications may be made to the process which comes within the scope of this instant invention.

What is claimed is:

1. The method of enclosing a gas within a solid matrix to yield a gas-containing solid which is substantially stable at room temperature for extended periods of time comprising gasifying a fused fusible sugar at superatmospheric pressure and cooling said gasified fused fusible sugar below its fusion temperature under said gasifying pressure thereby forming a product gasified sugar containing therewithin a gas under superatmospheric pressure.

2. The method claimed in claim 1 wherein said fusible sugar has a fusing point of 25° C.–200° C.

3. The method claimed in claim 1 wherein said gasifying is effected under superatmospheric pressure of 50–1000 p.s.i.g.

4. The method claimed in claim 1 wherein the gasifying is effected with a gas selected from the group consisting of carbon dioxide, nitrogen, and air.

5. The method claimed in claim 1 wherein said gasifying is for time sufficient to permit adsorption in said fused fusible sugar in 0.5–15 ml. of gas per gram of sugar.

6. The method claimed in claim 1 wherein said fusible sugar is in near anhydrous state.

7. The method claimed in claim 1 wherein said fusible sugar is selected from the group consisting of glucose, fructose, arabinose, sucrose, lactose, maltose, fructosan, gentiobiose, cellobiose, panose, malto-triose, malto-tetrose, sorbitol, mannitol, gluconic acid, and saccharic acid.

8. The method of enclosing a gas within a solid matrix to yield a gas-containing solid which is substantially stable

at room temperature for extended periods of time comprising fusing a fusible sugar, contacting said fused sugar with gas under superatmospheric pressure, maintaining the temperature of said fused sugar above its fusion temperature during the period of gas adsorption, and cooling said fused sugar under said gasifying pressure to a temperature below its fusion temperature thereby obtaining a gas-containing solid.

9. The method of enclosing a gas within a solid matrix to yield a gas-containing solid which is substantially stable at room temperature for extended periods of time comprising fusing a near-anhydrous fusible sugar, contacting said fused fusible sugar with gas at a pressure of 50–1000 p.s.i.g. to permit adsorption therein of 0.5–15 ml. gas per gram of sugar, maintaining said sugar under said gasifying pressure at temperature above its fusion point during said fused adsorption, and cooling said sugar to a temperature below its fusion temperature thereby obtaining a gas-containing solid.

10. A gas-containing solid characterized by its substantial stability at room temperature for extended periods of time and by its capacity to liberate gas contained therein comprising a solidified fused fusible sugar containing therewithin a gas under superatmospheric pressure.

11. A gas-containing solid characterized by its substantial stability at room temperature for extended periods of time and by its capacity to liberate gas contained therein comprising a substantially anhydrous solidified fused fusible sugar containing therewithin a gas under superatmospheric pressure.

12. A gas-containing solid characterized by its substantial stability at room temperature for extended periods of time and by its capacity to liberate gas contained therein comprising solidified fused sucrose containing therewithin a gas under superatmospheric pressure.

13. A gas-containing solid characterized by its substantial stability at room temperature for extended periods of time and by its capacity to liberate gas contained therein comprising solidified fused sucrose containing therewithin carbon dioxide under superatmospheric pressure.

14. A gas-containing solid characterized by its substantial stability at room temperature for extended periods of time and by its capacity to liberate gas contained therein comprising solidified fused sucrose and a material selected from the group consisting of dextrin, starch, gelatin, and gums, containing therewithin a gas under superatmospheric pressure.

15. A gas-containing solid characterized by its substantial stability at room temperature for extended periods of time and by its capacity to liberate gas contained therein comprising a solidified fused mixture of sucrose and corn syrup containing therewithin carbon dioxide under superatmospheric pressure.

16. A gas-containing solid characterized by its substantial stability at room temperature for extended periods of time and by its capacity to liberate gas contained therein comprising a solidified fused mixture of sucrose and lactose containing therewithin carbon dioxide under superatmospheric pressure.

17. A gasified confection characterized by its ability to liberate gas during dissolution in the mouth comprising a solidified fused sugar containing therewithin a gas under superatmospheric pressure.

18. A gasified confection characterized by its ability to liberate gas during dissolution in the mouth comprising a solidified fused sugar containing therewithin a gas under superatmospheric pressure and flavoring.

19. A carbonated solid characterized by a substantial stability at room temperature for extended periods of time, a fast rate of dissolution in water, a density heavier than water, and by liberation of large size bubbles when in contact with water comprising a solidified fused fusible sugar containing therewithin under superatmospheric pressure 0.5-5 ml. of carbon dioxide per gram of sugar.

20. A carbonated solid characterized by a substantial

stability at room temperature for extended periods of time, a slow rate of dissolution in water, a density lighter than water, and by liberation of initially large sized bubbles when in contact with water comprising a solidified fused fusible sugar containing therewithin under superatmospheric pressure 5–15 ml. of carbon dioxide per gram of sugar.

21. The method of enclosing a gas within a solid matrix to yield a gas-containing solid which is substantially stable at room temperature for extended periods of time comprising gasifying a fused fusible sugar under superatmospheric pressure and rapidly cooling said gasified fused sugar under said gasifying pressure below its fusion temperature.

22. The method of enclosing a gas within a solid matrix to yield a gas-containing solid which is substantially stable at room temperature for extended periods of time comprising gasifying a fused fusible sugar under superatmospheric pressure and cooling said gasified fused sugar under said gasifying pressure to below its fusion temperature, at a rate sufficiently rapid to minimize crystallization of said sugar.

23. The method of enclosing a gas within a solid matrix to yield a gas-containing solid which is substantially stable at room temperature for extended periods of time comprising gasifying a fused fusible near-anhydrous sugar under superatmospheric pressure and cooling said gasified fused fusible sugar under said gasifying pressure below its fusion temperature.

24. A gas-containing solid characterized by its substantial stability at room temperature for extended periods of time and by its capacity to liberate gas contained therein comprising a solidified fused fusible near-anhydrous sugar containing therewithin a gas under superatmospheric pressure.

25. The method of enclosing a gas within a solid matrix to yield a gas-containing solid which is substantially stable at room temperature for extended periods of time comprising gasifying a fused fusible sugar under superatmospheric pressure, cooling said gasified fused fusible sugar below its fusion temperature and maintaining said fused fusible sugar during said cooling at substantially the gasifying pressure.

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