(54) Title: MULTI-STAGE MEMBRANE CONCENTRATION METHODS AND PRODUCTS

(57) Abstract: A method is provided comprising passing a product through a first filtration membrane having a molecular weight cutoff (MWCO) greater than 500,000 Daltons using a pressure of between 10 pounds per square inches (psi) and 150 psi, thereby providing a first filtration retentate and a first filtration permeate. The first filtration permeate is passed through a second filtration membrane using a pressure of between 250 psi and 3,500 psi, thereby providing a second filtration retentate and a second filtration permeate. The second filtration retentate is passed through a third filtration membrane, using a pressure of between 250 psi and 3,500 psi, thereby providing a third filtration retentate and a third filtration permeate. Also, a concentrated product is provided having a brix of at least 35 degrees, for instance prepared by combining the first and third filtration retentates.
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MULTI-STAGE MEMBRANE CONCENTRATION METHODS AND PRODUCTS

CROSS-REFERENCE OF RELATED APPLICATIONS

[01] This application claims priority to U.S. Provisional Application 61/603,059 filed 24 February 2012, hereby incorporated by reference in its entirety.

FIELD OF THE INVENTION

[02] This invention relates to methods for multi-stage membrane concentration of products, concentrated products made using multi-stage membrane concentration methods, and reconstituted products. This invention further relates to multi-stage membrane selective separation of components from products.

BACKGROUND

[03] Methods for preparing products are desirable to achieve desired nutritional characteristics, flavor, shelf life, and other advantages. For example, an edible product is often subjected to concentration processes in order to decrease the water content and thereby decrease the volume and weight of the product for storage and transportation.

[04] One drawback to certain methods for concentrating products, for example thermal methods, is the loss of flavor and aroma compounds during the concentration processes. Other methods relate to membrane concentration of products; however, membranes which rely on concentration gradients and the diffusion of a solvent through a semi-permeable membrane are limited by the osmotic pressure of the final concentrated solute and subsequently the high pressures required to force the solvent through the membrane beyond the osmotic pressure of the solute, such as pressures of greater than 3,000 pounds per square inch (psi). The need for such high pressures can be impractical in certain applications.

[05] It would be desirable to provide a concentrated product having desirable nutritional and taste properties that is conveniently and practically prepared.
SUMMARY

In accordance with one aspect, a method is provided comprising passing a product through a first filtration membrane having a molecular weight cutoff (MWCO) less than or equal to 500,000 Daltons, or alternatively, having a pore size of between about 0.1 microns (\(\mu\)m) and 1.5 \(\mu\)m, using a pressure of between 10 pounds per square inches (psi) and 150 psi, thereby providing a first filtration retentate and a first filtration permeate. The first filtration permeate is passed through a second filtration membrane, the second filtration membrane optionally having an effective sugar rejection capability of between about 1% and about 99.9% sugar rejection, or alternatively, having a MWCO of between 100 and 10,000 Daltons, using a pressure of between 250 psi and 3,500 psi, thereby providing a second filtration retentate and a second filtration permeate. The second filtration retentate is passed through a third filtration membrane using a pressure of between 250 psi and 3,500 psi, thereby providing a third filtration retentate and a third filtration permeate. Optionally, the first filtration membrane comprises a hollow fiber, tubular, stainless steel, or ceramic filtration membrane and one or more of the second and third filtration membranes is selected from the group consisting of a spiral wound membrane, a hollow fiber membrane, a tubular membrane, a flat sheet plate and frame membrane, and a ceramic membrane.

In accordance with another aspect, a method is provided comprising passing a product through a first filtration membrane having a molecular weight cutoff (MWCO) less than or equal to 500,000 Daltons, or alternatively, having a pore size of between about 0.1 microns (\(\mu\)m) and 1.5 \(\mu\)m, using a pressure of between 10 pounds per square inches (psi) and 150 psi, thereby providing a first filtration retentate and a first filtration permeate. The first filtration permeate is passed through a second filtration membrane, the second filtration membrane optionally having an effective selectivity rate, in terms of sugar (e.g., combined monosaccharides, disaccharides, and polysaccharides) rejection, of between about 1% and 99.9% sugar rejection, or alternatively, having a MWCO of between 100 and 10,000 Daltons, using a pressure of between 250 psi and 3,500 psi, thereby providing a second filtration retentate and a second filtration permeate. The second filtration retentate is passed through a third filtration membrane, thereby providing a third filtration retentate and a third filtration permeate.
permeate. Each of the second filtration permeate and the third filtration permeate are returned and passed through the second filtration membrane to further concentrate the second and third permeates. At the conclusion of the multi-stage membrane concentration, the first filtration retentate is combined with the retentate of the final filtration stage to provide a concentrated product. Optional!), the first filtration membrane comprises a hollow fiber, tubular, stainless steel, or ceramic filtration membrane, and one or more of the second and third filtration membranes is selected from the group consisting of a spiral wound membrane, a hollow fiber membrane, a tubular membrane, a flat sheet plate and frame membrane, and a ceramic membrane.

In accordance with yet another aspect, a method is provided comprising passing a product through a first filtration membrane selected from the group consisting of a hollow fiber membrane, a tubular membrane, a stainless steel membrane, and a ceramic membrane, the first filtration membrane having a molecular weight cutoff (MWCO) greater than 500,000 Daltons, thereby providing a first filtration retentate and a first filtration permeate. The first filtration permeate is passed through a second filtration membrane using a pressure above and outside of the membrane manufacturer specification, thereby providing a second filtration retentate and a second filtration permeate; and the second filtration retentate is passed through a third filtration membrane using a pressure above and outside of the membrane manufacturer specification, thereby providing a third filtration retentate and a third filtration permeate. The first filtration permeate may be passed through the second filtration membrane at a pressure of between 250 psi and 3,500 psi. In certain aspects of the method, the second filtration membrane has an effective sugar rejection capability of between about 1% and about 99.9% sugar rejection. Optionally, the second filtration membrane is selected from the group consisting of a spiral wound membrane, a tubular membrane, a hollow fiber membrane, a flat sheet plate and frame membrane, and a ceramic membrane.

In accordance with a further aspect, a concentrated product is provided comprising a brix of at least 35 degrees brix, or at least 40 brix, or at least 55 brix, or at least 60 brix, or at least 65 brix, or at least 70 brix.

In a still further aspect, a product is provided comprising water and a concentrated product, where the product comprises a from concentrate 100% orange juice product
comprising a flavor and an aroma that are statistically identical to the flavor and the aroma of a not from concentrate orange juice, as determined by a sensory panel.

[11] In yet a further aspect, a product is provided comprising water and a concentrated product, where the concentrated product comprises a juice product comprising a brix of at least 65 degrees brix. The product comprises a 100% juice product comprising a flavor and an aroma that are statistically equal to or superior to the flavor and the aroma of the same juice prepared by combining water and a concentrated juice comprising the same brix and concentrated using thermal methods, as determined by a sensory panel.

[12] In a further aspect, a method is provided comprising passing a clarified product through a plurality of filtration membranes independently selected from the group consisting of a hollow fiber membrane, a tubular membrane, a spiral wound membrane, a stainless steel membrane, and a ceramic membrane, thereby providing at least one filtration retentate comprising a brix value of at least 65 brix and at least one filtration permeate. At least one of the plurality of filtration membranes comprises an ultrafiltration membrane or a nanofiltration membrane used at a pressure above and outside of the manufacturer specification.

[13] In another aspect, a method of selectively separating a component from a liquid-based composition is provided comprising passing a liquid-based composition that does not contain insoluble solids through a filtration membrane using a pressure of between 250 psi and 3,500 psi, thereby providing a first filtration retentate and a first filtration permeate. Next, passing the first filtration retentate through a second filtration membrane using a pressure of between 250 psi and 3,500 psi, thereby providing a second filtration retentate and a second filtration permeate, wherein the component comprises a molecular weight at least ten times smaller than the molecular weight cut-off of the second filtration membrane.

[14] It will be appreciated by those skilled in the art, given the benefit of the following description of certain exemplary embodiments of the methods and products disclosed here, that at least certain embodiments of the invention have improved or alternative formulations suitable to provide desirable taste profiles, nutritional characteristics, etc. These and other aspects, features and advantages of the invention or of certain
embodiments of the invention will be further understood by those skilled in the art from the following description of exemplary embodiments.

BRIEF DESCRIPTION OF THE DRAWINGS

[15] FIG. 1 shows a diagram of a multi-stage membrane filtration method according to an embodiment of the invention.

[16] FIG. 2 shows a theoretical depiction of the filtration of a liquid through a membrane.

[17] FIG. 3 shows a graph of percent rejection following filtration of a 40 brix simulated juice at various pressures, according to an embodiment of the invention.

[18] FIG. 4 shows a graph of percent rejection following filtration of a 40 brix simulated juice at various pressures, according to another embodiment of the invention.

[19] FIG. 5 shows a graph of percent rejection following filtration of a 50 brix simulated juice at various pressures, according to an embodiment of the invention.

[20] FIG. 6 shows a graph of percent rejection following filtration of a 50 brix simulated juice at various pressures, according to another embodiment of the invention.

[21] FIG. 7 shows a graph of percent rejection following filtration of a 60 brix simulated juice at various pressures, according to an embodiment of the invention.

[22] FIG. 8 shows a graph of glucose and sucrose content in feed solutions and permeate solutions, according to an embodiment of the invention.

[23] FIG. 9 shows a graph of percent rejection of linalool following filtration of a 37.5 brix simulated juice at various pressures, according to an embodiment of the invention.

DETAILED DESCRIPTION OF EMBODIMENTS

[24] Liquid-containing products, such as beverages, snacks and soups, are popular with consumers for numerous reasons, such as their nutrients, taste, and convenience. However, the presence of a significant amount of liquid can be undesirable when the product must be stored or transported prior to consumption by a user. It is an advantage of embodiments of the present invention to provide a practical multi-stage membrane method for concentrating a product. It is a further advantage of
embodiments of the present invention to provide a concentrated product that retains most or all of the nutrients, the flavor or aroma compounds, or combinations thereof. Similarly, it is an advantage of embodiments of the present invention to provide a reconstituted product comprising a liquid and a concentrated product, in which most or all of the nutritional, flavor and aroma characteristics of the original single-strength product are maintained. Moreover, it is further an advantage of embodiments of the present invention to selectively remove components from products using multi-stage membrane processes.

[25] A suitable filtration membrane for use in separating insoluble solids from a product prior to concentration is a hollow fiber membrane, a tubular membrane, a ceramic membrane, a stainless steel membrane, or combinations thereof, for example in series. Such membranes are well known in the art and are commercially available from suppliers such as SpinTek Systems (Los Alamitos, CA), Pall Corporation (Port Washington, NY), Atech Innovations GmbH (Gladbeck, Germany), TAMI Industries (Montreal, Canada), Graver Technologies (Glasgow, DE) and Koch Membranes (Wilmington, MA). As used herein, the term "insoluble solid" refers to any material that is in suspension as opposed to being in solution. The filtration membrane for separating insoluble solids, preferably thereby providing a clarified product, comprises either a molecular weight cutoff (MWCO) of 500,000 Daitons, or of greater than about 500,000 Daitons. Alternatively, the filtration membrane comprises a pore size of about 0.1 microns (μm), or a pore size between about 0.1 μm and 1.5 μm. Typically, the filtration to separate insoluble solids is performed using a pressure of between 10 pounds per square inches (psi) and 150 psi, such as between 20 psi and 100 psi, or about 50 psi. In an embodiment, more than one filtration membrane is used in series to clarify a product, for example a hollow fiber filtration membrane comprising a MWCO of 500,000 Da may be employed in the first stage, using a pressure of between 10 psi and 50 psi, followed by a ceramic or stainless steel type with a pore size between about 0.1 and 1.5 μm, using a pressure of up to 150 psi, to separate the insoluble solids from a product. In certain embodiments, insoluble solids are concentrated by up to a factor of ten using the first filtration membrane, for example a concentration factor between one and eight. For instance, in a particular product comprising 2% insoluble solids, following passing the product through the first filtration membrane, a first filtration retentate may be obtained comprising
between 4% and 14% insoluble solids. In certain embodiments, the insoluble solids are concentrated from a particular product from a starting insoluble solid amount of 5-8% up to between 50% and 100% insoluble solids.

[26] A suitable filtration membrane for use in embodiments of the invention is a spiral wound membrane. Spiral wound membranes are well known in the art of filtration, for example in methods for the desalination of water. A detailed description of spiral wound membrane may be found in the book "Ultrafiltration and Microfiltration Handbook", Munir Cheryan, CRC Press LLC, Boca Raton, FL (1998), pp. 210-226. Briefly, spiral wound membranes comprise at least one pair of flat membrane sheets separated by one or more spacers, which form a permeate channel. The pair(s) of membrane sheets are affixed together around their edges and rolled into a tube shape around a perforated center collection tube, then inserted into a tubular housing. In use, a feed solution is pumped lengthwise into one end of the housing, along the membrane, and the permeate passes through the membrane sheets into the spiral permeate channel until it reaches the center collection tube. The retentate is collected via an outlet in the housing. Spiral wound filtration membranes are typically operated at pressures of between about 60 psi (4 bar) and about 400 psi (28 bar). The pressure drop in spiral wound membranes is typically limited to about 10-20 psi (0.7-1.4 bar), in order to prevent damaging the membranes.

[27] Another suitable filtration membrane for use in embodiments of the invention is a tubular membrane. Tubular membranes are well known in the art of filtration and a detailed description of tubular membranes may be found in the book "Ultrafiltration and Microfiltration Handbook", Munir Cheryan, CRC Press LLC, Boca Raton, FL (1998), pp. 178-190. Briefly, tubular membranes comprise polymeric membranes cast on the inside surfaces of inserts composed of plastic or paper. Typically, a plurality of tubes are assembled and encased in a housing. For use in reverse osmosis applications, a feed solution is passed through the plurality of tubes in series, while for use in ultrafiltration or microfiltration applications a feed solution is passed through the plurality of tubes in parallel; the retentate is collected via an outlet in the housing. Tubular membranes may be manufactured comprising a nominal molecular weight cutoff (MWCO) of as small as 10,000 Daltons to as large as 250,000 Daltons. Alternatively, tubular membranes may comprise a pore size of as low as about 0.3
microns up to about 2-3 microns. Tubular membranes are typically operated at pressures up to about 150 psi (10.5 bar). Tubular membranes prepared such that the polymer is cast on the insert while it is manufactured in a helical-spiral configuration, however, are typically operated at pressures up to as high as 450 psi.

[28] A further suitable filtration membrane for use in embodiments of the invention is a hollow fiber membrane. Hollow fiber membranes are well known in the art of filtration and a detailed description of hollow fiber membranes may be found in the book "Ultrafiltration and Microfiltration Handbook", Munir Cheryan, CRC Press LLC, Boca Raton, FL (1998), pp. 190-201. Briefly, hollow fiber membranes comprise polymeric membranes manufactured as self-supporting tubes. Similar to tubular membranes, a plurality of tubes are assembled and encased in a housing. For use in reverse osmosis applications, a feed solution is passed from the exterior of the tubes and permeate is removed through the inside of the tubes. For use in ultrafiltration or microfiltration applications a feed solution is passed through the inner core of tubes and the permeate is collected via outlets at either end of the housing. Hollow fiber membranes may be manufactured comprising a nominal molecular weight cutoff (MWCO) of as small as 1,000 Daltons to as large as 500,000 Daltons. Alternatively, hollow fiber membranes may comprise a pore size of as low as about 0.1 microns up to about 0.85 microns. Hollow fiber membranes are typically operated at pressures up to about 145 psi (10 bar), except for reverse osmosis membranes operated at pressures of 600-900 psi.

[29] An additional suitable filtration membrane for use in embodiments of the invention is a flat sheet plate and frame membrane. Flat sheet plate and frame membranes, also referred to as plate-type membranes, are well known in the art of filtration and a detailed description of plate-type membranes may be found in the book "Ultrafiltration and Microfiltration Handbook", Munir Cheryan, CRC Press LLC, Boca Raton, FL (1998), pp. 202-210. Briefly, flat sheet plate and frame membranes comprise a flat polymeric membrane placed on a net-like material on one side of a plate. On the opposite of the plate another permeate spacer and polymeric membrane sheet are placed, and the sheets are typically sealed around the edges but have an outlet for the permeate. A plurality of plate-type membranes may be arranged in a vertical or horizontal stack to form a module or cartridge. Plate-type membranes may
be manufactured comprising a nominal molecular weight cutoff (MWCO) of as small as 1,000 Daltons to as large as 500,000 Daltons. Alternatively, plate-type membranes may comprise a pore size of as low as about 0.1 microns up to about 2 microns. Flat sheet plate and frame membranes are typically operated at pressures of about 29-145 psi (2-10 bar), occasionally up to about 580 psi (40 bar).

Another suitable filtration membrane for use in embodiments of the invention is a ceramic membrane. Ceramic membranes are well known in the art of filtration and a detailed description of ceramic-type membranes may be found in the book "Ultrafiltration and Microfiltration Handbook", Muñir Cheryan, CRC Press LLC, Boca Raton, FL (1998), pp. 57-68. Briefly, ceramic membranes comprise inorganic materials, and are usually available as single-channel tubes or multichannel tubular elements. A macroporous substrate is formed by heating an extruded paste of one or two inorganic powders, followed by coating the inside of the membrane channels with a fine-grain powder. Pressurizing, drying and baking steps are employed to set the membrane. Single tubes may be bundled and placed in a stainless steel housing. In use, a feed solution is passed through the inside of the channels and the permeate flows through a support layer around the tubes to the outside of the membrane. Ceramic membranes may be manufactured comprising a nominal molecular weight cutoff (MWCO) of as small as 15,000 Daltons to as large as 300,000 Daltons. Alternatively, ceramic membranes may comprise a pore size of as low as about 0.1 microns up to about 1 micron. Ceramic membranes are typically operated at pressures up to about 145 psi (10 bar).

The pore size of a filtration membrane typically dictates what size of components will be retained by the membrane or will pass through the membrane; thus a membrane having a molecular weight cutoff (MWCO) of greater than about 200,000 Daltons (Da) and less than or equal to about 500,000 Daltons allows smaller compounds (e.g., comprising a molecular weight of less than 5,000 Daltons) to pass through the much larger pores. Such membranes are advantageously employed in embodiments of the invention to separate insoluble solids from compositions in order to provide a clarified solution or serum to concentrate in the multiple stages. Another way to define the filtration capabilities of a filtration membrane is by its salt rejection, which refers to the percentage of salt ions, such as magnesium sulfate ions, that are rejected by the
membrane. For example, filtration membranes employed in aspects of the invention optionally have a salt rejection capability of between about 1% and about 99.9% magnesium sulfate rejection. A salt rejection capability of between about 10% and 99.9% magnesium sulfate rejection is referred to as a "low rejection membrane", and a salt rejection capability of between about 90% and 99.9% magnesium sulfate rejection is referred to as a "high rejection membrane". U.S. Patent 4,959,237 discloses that when separating solids from serum using an ultrafiltration membrane comprising pores of less than or equal to 200,000 Da MWCO, aroma and flavor components are retained in the filtration retentate stream. In contrast, it has unexpectedly been discovered that separating solids from serum can be accomplished with membranes comprising a MWCO in between about 200,000 and 500,000 Da, or membranes comprising a MWCO of greater than 500,000 Da, or membranes having a pore size of up to about 1.5 μm.

[32] For example, a membrane comprising a MWCO of 500,000 Da is expected to allow smaller compounds, such as pectin methyl esterase (PME) having a size between approximately 34,000 Da and approximately 36,000 Da, to pass through; however, when juice products comprising PME are subjected to filtration according to at least certain embodiments, the PME is retained with the solids fraction. Without washing to be bound by theory, this is likely due to adherence of PME to the insoluble solids matrix. Consequently, if a clarified juice serum is produced by filtration through such a membrane, then PME as well as any other compounds that are entrained within the solids matrix are retained. In contrast, employment of a filter comprising a pore size of 5 μm, for instance, for insoluble solids separation would not produce clarified juice serum in the permeate and PME would likely be present in the filtration permeate.

[33] As noted above, the pore size of a filtration membrane typically dictates what size of components will be retained by the membrane or will pass through the membrane; thus a membrane having a molecular weight cutoff of between about 1,000 Daltons and about 5,000 Daltons would be expected to allow smaller compounds (e.g., comprising a molecular weight significantly less than 5,000 Daltons) to pass through the much larger pores. It was therefore a surprising discovery of embodiments of the present invention that it is possible to retain components having low molecular weights in a liquid-based product without requiring the use of a membrane having a
pore size equal to or smaller than the desired components. In particular, it was discovered that a membrane can be used to filter a product and successfully retain a component between about 10 and 40 times smaller than the pore size when used at a pressure significantly outside of the manufacturer's recommended and typical operating pressure. In certain embodiments a component retained by the filtration membrane comprises a molecular weight at least ten times smaller than the MWCO of the filtration membrane, or at least twenty times smaller than the MWCO of the filtration membrane, or at least thirty times smaller than the MWCO of the filtration membrane. For instance, a membrane comprising a MWCO of at least 1,000 Daltons is generally operated at a pressure of between about 100 psi and 200 psi. In contrast, when the membrane is instead operated at a pressure of between 250 psi and 3,500 psi, or at least 700 psi, or at least 800 psi, a greater number of small components, such as between about 100 and 400 Daltons in size, are retained.

[34] Without wishing to be bound by theory, it is believed that the high pressure causes both compression of the membrane, thereby altering the membrane morphology and decreasing flux, and an increase of the concentration polarization layer present at the surface of the membrane, which together greatly enhance the rejection capabilities of the membrane. Although the phenomenon of the formation of a concentration polarization layer at a filtration membrane, which acts as a secondary boundary layer for solute transport, has previously been identified, it was an unexpected discovery that such a large increase in rejection could be achieved by employing filtration membranes at pressures above and outside of the manufacturer specifications, such as between about 250 psi and 3,500 psi. For example and without limitation, the increase in sugar rejection according to at least certain embodiments comprises ten to twenty times the percent rejection achieved at typical pressures (e.g., less than about 250 psi).

[35] A concentration polarization layer, i.e., a secondary membrane formed on the surface of the membrane, is a layer of components from the feed, for instance sugars in a juice feed. The secondary boundary layer is optionally referred to as a "gel" layer or concentration polarization layer. The characteristics of a secondary boundary layer formed on the surface of the membrane are controlled by several factors of the filtration process, including feed solute concentration, temperature, pressure, and
cross-flow velocity. For example, assuming all other conditions remain the same, an increase in temperature of the process will decrease the viscosity of the secondary boundary layer, whereas a decrease in the temperature of the process will increase the viscosity of the secondary boundary layer. A more viscous layer will result in greater solute rejection, whereas a less viscous layer will result in lower solute rejection. As the operating pressure of a filtration process is increased, the bulk transport of solutes to the surface of the membrane also increases, which results in an increase in the concentration at the surface of the membrane and adds to the thickness of the boundary layer. Moreover, compression of the polarization layer at higher pressures also causes an increase in the concentration of solutes, for instance sugars, in the secondary boundary layer. Similarly, assuming all other conditions remain the same, as the cross-flow velocity is increased, the thickness of the secondary boundary layer decreases, whereas a decrease in the cross-flow velocity will increase the thickness of the secondary boundary layer. The maximum potential cross-flow velocity will typically be determined by practical considerations such as the amount of pressure drop that a particular filtration membrane can withstand without becoming damaged. Alternatively, additional processing techniques can affect the secondary boundary layer, for instance subjecting the filtration membrane to agitation may have the effect of minimizing buildup of a secondary boundary layer at the surface of a membrane. Accordingly, careful manipulation of processing parameters such as pressure, temperature, cross-flow velocity and feed concentration, according to at least certain embodiments disclosed herein, may allow the skilled practitioner to generate a polarization layer capable of achieving high rejections acceptable for the multi-rejection membrane processes disclosed here.

[36] According to certain embodiments, the concentration polarization layer comprises a greater concentration of sugars than in the bulk solution, which results in effective increasing the sugar rejection of the membranes employed at pressures between about 250 psi and about 3,500 psi. For example, the effective sugar rejection, including sugars selected from monosaccharides, disaccharides, polysaccharides and combinations thereof, comprises a sugar rejection of between about 1% and 99.9% sugar rejection. In certain aspects, the effective sugar rejection comprises between about 10% and 90% sugar rejection, or between about 10% and 99.9% sugar rejection, or between about 90% and 99.9% sugar rejection.
[37] The selectivity of the membrane, with respect to at least water soluble components (for example and without limitation sugars and volatile flavor compounds), improves as the operating pressure increases, such as between about 250 psi and no more than about 3,500 psi, above which little further selectivity is achieved. According to certain aspects of the invention, the operating pressure is between about 250 psi and about 3,500 psi, or between about 400 psi and about 3,000 psi, or between about 500 psi and about 2,500 psi, or between about 700 psi and about 2,200 psi, or between about 800 psi and about 1,700 psi, or between about 850 psi and about 1,600 psi, or between about 900 psi and about 1,500 psi, or between about 1,000 psi and about 1,400 psi. The pressure is provided by any suitable pressure source, such as a pump typically employed in the membrane filtration art, or by manipulation of other apparatus components, for instance via restricting flow downstream of the filtration membranes. Such manipulation optionally comprises partially or completely closing apparatus valves located downstream of the filtration processes, thereby increasing the pressure of the processes up to the pump. In certain aspects, pressures of up to 3,500 psi are obtained using a pump operating at a pressure of between 50 and 100 psi, through the use of line constriction within the apparatus.

[38] A variety of filtration membranes are commercially available in the art, and are typically referred to as ultrafiltration (UF) membranes, nanofiltration (NF) membranes, reverse osmosis (RO) membranes, and the like. Each type of membrane has its own characteristic pore size ranges or magnesium sulfate salt rejection percentage ranges. It is an advantage of at least certain aspects that the manipulation of processing parameters, such as pressure, is capable of achieving solute rejection using a membrane having much larger pores, which is typically only achieved with a membrane having much smaller pores. For example, it was unexpectedly discovered that the rejection capability of UF membranes operating at very high pressures approaches rejection capability of NF membranes operating at standard, low pressures. Similarly, it may be possible to employ a ceramic membrane, having a MWCO of about 1000 Da, using low cross-flow velocity, low temperature, high concentration, and very high pressures, to form a polarization layer on the surface of the membrane that results in flux and rejection characteristics suitable for multi-stage membrane separation processes.
Without wishing to be bound by theory, the concentration data of sugar solutions having a brix above 35 brix using high pressures, combined with transport models, indicate that this high rejection phenomenon is likely due primarily to concentration polarization effects for sugar solutions having non-dilute concentrations. Considering that many filtration membranes have a manufacturer's specification for use at pressures of about 250 psi or less, it was a surprising discovery that many filtration membranes, including ultrafiltration membranes, nanofiltration membranes and reverse osmosis membranes, are capable of operating at extremely high pressures without being destroyed; for instance it was discovered that such membranes are sufficiently robust to be successfully employed for filtration processes using pressures as high as about 1,000 psi, about 1,500 psi, about 2,500 psi, or even about 3,500 psi.

The discovery of the capability of significantly increasing the rejection of a membrane to concentrate a solution can also be extended to the general principle that applying a pressure above and outside the manufacturer's operational pressure will selectively separate specific components from a composition. Accordingly, an embodiment of the invention comprises a method for selectively separating a component from a liquid-based composition that does not contain insoluble solids. The method comprises passing a liquid-based composition lacking insoluble solids through a filtration membrane using a pressure of between 250 psi and 3,500 psi, thereby providing a first filtration retentate and a first filtration permeate. The filtration membrane optionally has a sugar rejection capability when employed under conditions according to embodiments of the invention of between about 1% and about 99.9% sugar rejection (i.e., either a low rejection membrane or a high rejection membrane), for instance a sugar rejection capability of between about 10% and 99.9% sugar rejection, or between about 90% and 99.9% sugar rejection. The first filtration retentate is passed through a second filtration membrane using a pressure of between 250 psi and 3,500 psi, thereby providing a second filtration retentate and a second filtration permeate. The second filtration membrane optionally has a sugar rejection capability of between about 1% and about 99.9% sugar rejection, or, alternatively, a MWCO between about 100 and about 10,000 Daltons. In an embodiment, the pressure is chosen to achieve rejection of one or more selected components into the filtration retentates. In an alternate embodiment, the pressure is chosen to allow one or more selected components to pass into the filtration permeates.
As discussed above, the use of filtration membranes under conditions different from the manufacturer specifications, such as above and outside of the pressure specifications, impacts the sugar rejection capability of each filtration membrane. Accordingly, when the sugar rejection of a filtration membrane is referred to herein in a particular context, it should be noted that the sugar rejection percentage comprises the effective percentage of sugar rejection for that specific use - either within or outside of manufacturer's specifications. For example, a filtration membrane having a sugar rejection of less than 10% when employed at a pressure of about 100 psi and a selected temperature and cross-flow velocity may exhibit a sugar rejection of between 50% and 60% when employed at a pressure of about 1,000 psi and the same temperature and cross-flow velocity, thus the effective sugar rejection for a filtration membrane is in part a function of the filtration operating conditions.

In accordance with yet another aspect, a method is provided comprising passing a product through a first filtration membrane selected from the group consisting of a hollow fiber membrane, a tubular membrane, a stainless steel membrane, and a ceramic membrane, the first filtration membrane having a molecular weight cutoff (MWCO) greater than 500,000 Daltons, thereby providing a first filtration retentate and a first filtration permeate. The first filtration permeate is passed through a second filtration membrane using a pressure above and outside of the membrane manufacturer specification, thereby providing a second filtration retentate and a second filtration permeate, and the second filtration retentate is passed through a third filtration membrane using a pressure above and outside of the membrane manufacturer specification, thereby providing a third filtration retentate and a third filtration permeate. The first filtration permeate may be passed through the second filtration membrane at a pressure of between 250 psi and 3,500 psi. In certain aspects of the method, the second filtration membrane has an effective sugar rejection capability of between about 10% and about 99.9% sugar rejection. Optionally, the second filtration membrane is selected from the group consisting of a spiral wound membrane, a tubular membrane, a hollow fiber membrane, a flat sheet plate and frame membrane, and a ceramic membrane. One example of selectively separating components comprises a liquid-based composition comprising a mix of saccharides, in which disaccharides are selectively retained in the filtration retentates as compared to monosaccharides.
It was a further discovery that methods according to at least certain embodiments are capable of concentrating solutions containing small, water-soluble volatile compounds present in low concentrations, while retaining a significant amount of these small compounds. Such small, water-soluble volatile compounds include for example and without limitation, aldehydes, esters, alcohols, terpene hydrocarbons, ketones, and compounds associated with off-notes, which are typically found in juice, such as associated with flavors. For example and without limitation, such compounds include acetyldehyde, hexanal, trans-2-hexenal, octanal, nonanai, citronellal, decanal, undecanal, neral, dodecanal, geranial, beta-sinensal, alpha-sinensal, methyl butyrate, ethyl butyrate, ethyl 2-methylbutyrate, ethyl caproate, ethyl 3-hydroxyhexanoate, ethyl alcohol, linalool, octanol, decanol, citronellol, nerol, geraniol, alpha-pinene, myrcene, limonene, valencene, ethyl vinyl ketone, nootkatone, 2-pentanone, p-cymene, 1-hexanol, cis-3-hexenol, terpinen-4-ol, alpha-terpineol, carvone, trans-carveol, cis-carveol, beta-ionone, 4-vinyl-2-methoxy-phenol, and 4-vinylphenol. Such compounds are often added to juice, as a so-called “add back” to replace small volatile compounds that can be lost during processing, storage, or combinations thereof, of the juice.

Small, water-soluble volatile compounds have a size of approximately 50-200 Daltons, and are typically present in juice in an amount of approximately 50-300 ppm each. Accordingly, it was a surprising discovery that it is possible to achieve significant retention of a small, water-soluble and volatile compound in a liquid product at filtration pressures well above and outside the membrane manufacturer's established operating pressure range.

As discussed above, any suitable filtration membrane may be employed, for example spiral wound membranes. Various suitable membranes are commercially available from Koch Membranes (Wilmington, MA), Synder Filtration (Vacaville, CA), Sepro Membranes, Inc. (Oceanside, CA), Dow Chemical Company (Midland, MI), General Electric Company (Fairfield, CT), and Hydranautics (Oceanside, CA). NF6 - Sepro, VT - Synder, and NTR-7470 - Hydranautics are a few specific membranes useful for embodiments of the application. Each filtration membrane employed in the methods disclosed herein comprises a material independently selected from an inorganic material or a polymeric material. Suitable materials include for example and without
limitation, polystyrene, polyvinylidene fluoride, polytetrafluoroethylene, polyethersulfone, thin film composites, polyolefins, fluoropolymers, cellulose acetate, regenerated cellulose and combinations thereof.

In an embodiment, a method is provided comprising passing a product through a first filtration membrane having a MWCO greater than 500,000 Daltons, or alternatively, having a pore size of between about 0.1 μm and 1.5 μm, using a pressure of between 10 psi and 150 psi, thereby providing a first filtration retentate comprising insoluble solids and a first filtration permeate; and passing the first filtration permeate through a second filtration membrane having a sugar rejection capability of between about 1% and about 99.9%, or alternatively, having a MWCO of between 100 and 10,000 Daltons, using a pressure of between 250 psi and 3,500 psi, thereby providing a second filtration retentate and a second filtration permeate.

In certain aspects, the method further comprises combining the first and second filtration retentates to form a concentrated product having at least a minimum brix level. The concentrated product comprises a brix of greater than 25 degrees brix, such as 30 degrees brix or greater, 35 degrees brix or greater, 38 degrees brix or greater, 42 degrees brix or greater, 48 degrees brix or greater, 52 degrees brix or greater, 56 degrees brix or greater, 60 degrees brix or greater, or 65 degrees brix or greater, or 70 degrees brix or greater. The brix achieved via the multi-stage membrane concentration comprises two or more stages; accordingly, the skilled practitioner will understand that the method optionally comprises any number of additional filtration stages. In certain aspects, the second filtration retentate comprises a concentrated serum and the method further comprises passing the second filtration retentate through a third filtration membrane, thereby providing a third filtration retentate and a third filtration permeate. The third filtration retentate comprises a concentrated serum. Embodiments of the method further comprise passing the third filtration retentate through a fourth filtration membrane, thereby providing a fourth filtration retentate (i.e., concentrated serum) and a fourth filtration permeate. Similarly, embodiments of the method further comprise passing the fourth filtration retentate through a fifth filtration membrane, thereby providing a fifth filtration retentate (i.e., concentrated serum) and a fifth filtration permeate.
In exemplary aspects of the invention, a multi-stage membrane process advantageously provides for recycling of the filtration permeate from one or more of the membrane filtrations to further recover solute from the one or more permeates that had not been rejected by the filtration membrane. For an embodiment in which at least three stages are included, this may be accomplished by passing the second filtration permeate and the third filtration permeate through the second filtration membrane.

In at least certain aspects, a filtration permeate from a membrane filtration comprising a low brix, for instance less than about 10 degrees brix, or less than about 5 degrees brix, or about 2 degrees brix, or about 1 degree brix, or even as low as 0.05 brix. Prior to recycling such a low brix filtration permeate, the permeate is optionally passed through a filtration membrane to increase the permeate brix to about 10 degrees brix or higher, for instance as high as 63 brix. In an aspect, the filtration membrane is a high rejection membrane, such as a high rejection reverse osmosis membrane.

The concentrated product is formed by combining at least two filtration retentates together, for example combining each of the first and second filtration retentates, or each of the first and third filtration retentates to form a concentrated product. According to embodiments comprising four stages, the method optionally comprises combining each of the first and fourth filtration retentates to form a concentrated product. Likewise, the method optionally comprises combining each of the first and fifth filtration retentates to form a concentrated product.

The liquid-based product concentrated by a method according to embodiments of the invention is not particularly restricted. For example and without limitation, the product comprises one or more fruit juices, one or more vegetable juices, a soup, a liquid snack, or a beverage product selected from single strength beverage products, finished beverages and partially finished beverages. For concentrating any of these liquid-based products that contain large solid particulates (e.g., pieces of vegetables, grains and/or meats in a soup), it would be advantageous to first screen out the particulates. Further liquid-based products for concentration by methods of the present invention will be apparent to the skilled practitioner having benefit of the disclosure herein.
In embodiments of the invention, certain liquid-based products require destruction of microorganisms, for instance via pasteurization or other commercial sterilization methods. It is an advantage of at least some aspects of the invention that once the liquid-based product is pasteurized prior to passing the product through the first filtration membrane, it is not necessary to further sterilize the concentrated product. Alternatively, in other aspects, the methods further comprise pasteurizing the resulting concentrated product after the final filtration (e.g., after the second, third, fourth, fifth, etc., filtration).

In embodiments of the invention, a method is provided employing a combination of at least one high rejection membrane and at least one low rejection membrane. As used herein, the term "high rejection" refers either to a membrane that prevents at least 90% of magnesium sulfate ions from passing through membrane into the filtration permeate, or to a membrane that has a molecular weight cutoff of between about 100 Daltons and about 300 Daltons. Typically, rejection of a filtration membrane is referred to in terms of salt rejection, which provides a standard point of reference. In embodiments of the invention, high rejection membranes have a salt rejection capability of between about 90% and about 99.9% magnesium sulfate rejection. In embodiments of the invention, high rejection membranes prevent between about 90% and about 99.9% of sugars, monosaccharides, disaccharides and polysaccharides, present in the product from passing through the membrane into the filtration permeate. In contrast, as used herein, the term "low rejection" refers to a membrane that prevents 90% or less of the magnesium sulfate ions present in the product from passing through the membrane into the permeate, or to a membrane that prevents about 90% or less of the sugars present in the product from passing through the membrane into the permeate, or to a membrane that has a molecular weight cutoff of greater than 300 Daltons to 10,000 Daltons. In embodiments of the invention, low rejection membranes have a sugar rejection capability of between about 1% and about 90% sugar rejection, or between about 10% and about 90% sugar rejection, such as about 50% sugar rejection.

In certain exemplary aspects of the invention, various different arrangements of high rejection and low rejection filtration membranes are employed. For instance, in an embodiment the second filtration membrane is a high rejection membrane having a
sugar rejection capability of between about 90% and about 99.9% sugar rejection, and the first filtration permeate is passed through the second filtration membrane at a pressure between 250 psi and 3,500 psi. The third filtration membrane is a low rejection membrane having a sugar rejection capability of between about 1% and about 90% sugar rejection, such as between about 10% and about 90% sugar rejection. In an alternate embodiment, the second filtration membrane is a low rejection membrane having a sugar rejection capability of between about 1% and about 90% sugar rejection (e.g., between about 10% and about 90% sugar rejection) and the third filtration membrane is a high rejection membrane having a sugar rejection capability of between about 90% and about 99.9% sugar rejection. In general, for a multi-stage membrane process, any arrangement of high rejection and low rejection membranes may employed; however, the filtration of a solution comprising a brix of 50 degrees brix or higher through a high rejection membrane provides extremely slow filtration, thus for such practical reasons it may optional! be avoided.

The method optionally comprises passing a product through a first filtration membrane having a MWCO greater than 500,000 Daltons, or alternatively, having a pore size of between about 0.1 μm and 1.5 μm, using a pressure of between 10 pounds per square inches (psi) and 150 psi, thereby providing a first filtration retentate and a first filtration permeate. The first filtration retentate comprises most or all of the insoluble solids present in the original product, and the first filtration retentate is preferably recombined with a final filtration retentate to form a concentrated product. The first filtration permeate is passed through a second filtration membrane, having a sugar rejection rating of greater than 90%, or alternatively, having a MWCO of between 100 and 10,000 Daltons, using a pressure of at least 700 psi, where the second filtration membrane rejects 90% or more of sugars in the first filtration permeate, thereby providing a second filtration retentate and a second filtration permeate. The second filtration retentate is passed through a third filtration membrane, where the second filtration membrane rejects less than 90% of sugars in the second filtration retentate, thereby providing a third filtration retentate and a third filtration permeate. The method optionally comprises combining each of the first and third filtration retentates to form a concentrated product.
Referring to the figures, wherein like numbers refer to like features, FIG. 1 shows a diagram of a filtration method according to an embodiment of the invention. As illustrated in FIG. 1, a juice comprising a brix of 11 degrees and 5% by weight insoluble solids is passed 10 through an ultrafiltration membrane at a pressure between about 50 psi and 150 psi, such as a membrane comprising a MWCO of less than or equal to 500,000 Daltons, or a pore size of about 0.1 microns. The retentate from the ultrafiltration, having a brix of 11 degrees brix and a solids content of 10-80% by weight insoluble solids, is separated from the permeate and stored 20. The ultrafiltration penneate is passed 30 through a high rejection reverse osmosis filtration membrane, such as a thin film composite membrane with sugar rejection capabilities exceeding 90% sugar rejection. The high rejection reverse osmosis penneate comprises a low brix, such as less than 2 degrees brix, and is optionally referred to as a "water stream," although it typically contains some sugars and also some acid. This second filtration permeate is shown to be recycled back 35 to the second filtration membrane. The reverse osmosis retentate (i.e., second filtration retentate) comprises concentrated serum, and is shown in FIG. 1 to have a brix of about 35 degrees brix.

Next, the high rejection reverse osmosis retentate is passed 40 through a low rejection filtration membrane (i.e., a third filtration membrane). The low rejection, third, filtration membrane has an effective sugar rejection capability of between about 1% and about 90% sugar rejection, or has a MWCO of between about 1,000 and 10,000 Da. The low rejection penneate has a brix of about 28 degrees brix and is sent back 45 through the high rejection reverse osmosis membrane to collect any compounds that were passed through the membrane due to its low rejection characteristics, to be further concentrated.

The multi-stage membrane method illustrated in FIG. 1 diagrams a step-wise progression of passing sequential filtration retentates through low rejection filtration membranes to achieve increasingly higher brix filtration retentates. In particular, in the embodiment shown in FIG. 1, the third filtration retentate has a brix of about 42 degrees brix and is passed 50 through a fourth (low rejection) filtration membrane at a pressure between 800 psi and 1,400 psi. The fourth filtration permeate has a brix of about 35 degrees brix and is sent back 55 through the high rejection reverse osmosis membrane to be further concentrated. The fourth filtration retentate has a brix of
about 49 degrees brix and is passed 60 through a fifth (low rejection) filtration membrane at a pressure between 800 psi and 1,400 psi. The fifth filtration permeate has a brix of about 42 degrees brix and is sent back 65 through the high rejection reverse osmosis membrane to be further concentrated. The fifth filtration retentate has a brix of about 56 degrees brix and is passed 70 through a sixth (low rejection) filtration membrane at a pressure between 800 psi and 1,400 psi. The sixth filtration permeate has a brix of about 49 degrees brix and is sent back 75 through the high rejection reverse osmosis membrane to be further concentrated. The sixth filtration retentate has a brix of about 63 degrees brix and is passed 80 through a seventh (low rejection) filtration membrane at a pressure between 800 psi and 1,400 psi. The seventh filtration permeate has a brix of about 56 degrees brix and is sent back 85 through the high rejection reverse osmosis membrane to be further concentrated.

The seventh low rejection reverse osmosis retentate has a brix of greater than 65 degrees brix and is then combined with the stored 20 ultrafiltration solids retentate to form a blended concentrate comprising a brix of 60 degrees or greater. In the embodiment illustrated in FIG. 1, the concentrated product comprises a brix of 65 degrees brix and 7% by weight insoluble solids.

Accordingly, in certain embodiments of the invention, the filtration method comprises any number of filtration steps employing high rejection and low rejection filtration membranes. For instance, optionally, the method comprises passing the third filtration retentate through a fourth filtration membrane, thereby providing a fourth filtration retentate and a fourth filtration permeate, and combining each of the first and fourth filtration retentates to form a concentrated product. In an aspect, the method comprises passing the fourth filtration retentate through a fifth filtration membrane, thereby providing a fifth filtration retentate and a fifth filtration permeate, and combining each of the first and fifth filtration retentates to form a concentrated product.

In certain embodiments, the method comprises a batch operation, in which a liquid-based product is pumped from a feed tank and passed through a filtration membrane. The filtration permeate is typically returned to the feed tank for recycling to achieve further separation. In another embodiment, the method comprises a so-called "feed and bleed" continuous operation, in which principles of a batch operation and a single
pass operation are combined. Typically, for feed and bleed processes, the liquid-based product is passed through a filtration membrane using a first pump, and then a second pump is employed to maintain a recirculation loop to prevent any loss of pressure or flow rate. Once the pressures and flow rates are stabilized, retentate is bled off the recirculation loop as new feed product is pumped into the loop. In contrast, in further embodiments of the inventive method, a single pump is employed to drive the entire multi-stage system. In such aspects, the pressure for each sequential filtration step is measurably less than the pressure for the prior step, because there is a small pressure drop between the inlet and outlet of each filtration membrane. A typical pressure drop is about 10-20 psi, as noted above. Advantageously, although higher filtration pressures are employed than the membranes were designed to be operational, concentrated products are obtained using methods of the present invention in which pressures do not exceed about 3,500 psi.

A concentrated product according to aspects of the invention may subsequently be diluted with any suitable liquid. For instance, the concentrated product is diluted with water, in certain aspects. When the product comprises a juice, a product is optionally provided comprising water and a concentrated juice product, wherein the concentrated juice product comprises a brix of at least 42 degrees brix or at least 60 degrees brix. The product comprises a 100% juice product comprising a flavor and an aroma that are statistically equal to or superior to the flavor and the aroma of the same juice prepared by combining water and a concentrated juice comprising the same brix and concentrated using thermal methods, as determined by a sensory panel. When the product comprises orange juice, for example, the product is preferably diluted with water to form a from concentrate 100% orange juice product comprising a flavor and an aroma that are statistically equal to or superior to the flavor and the aroma of a from concentrate orange juice comprising the same brix and in which the orange juice was thermally concentrated, as determined by a sensory panel. Similarly, when the product comprises orange juice, the concentrated product is preferably diluted with water to form a from concentrate 100% orange juice product comprising a flavor and an aroma that are statistically identical to the flavor and the aroma of a not from concentrate orange juice, as determined by a trained sensory panel.
Sensory panels are well known to those of skill in the art, and comprise testers trained to evaluate the organoleptic attributes of comestibles, for instance by describing products on the basis of selected senses, such as taste, scent, feel, sound, and appearance. Individual attributes evaluated optionally include a variety of aroma, flavor and texture characteristics. Each attribute is evaluated either in comparison to a standard on a numerical scale (e.g., 0-15) or relative to other test samples. The numerical values assigned to each attribute may be statistically analyzed to determine if an attribute of one sample is statistically different from the same attribute in another sample.

An advantage of at least certain aspects of the present invention comprises a greater retention of components including for example and without limitation, vitamins, phytonutrients such as flavonoids, carotenoids, semi-volatile components, and volatile components than other methods of product concentration, such as thermal concentration. Such components may be measured in the liquid-based products, filtration permeates, and filtration retentates using gas chromatography mass spectrometry (GC-MS) and/or high pressure liquid chromatography (HPLC). Typically, semi-volatile components and volatile components comprise a size of between about 50 and 100 Da. In certain embodiments, the semi-volatile components and volatile components comprise one or more flavor compounds, aroma compounds, or combinations thereof. Without wishing to be bound by theory, it is believed that significant improvement in flavor retention as compared to thermal concentration methods is achieved when a product is concentrated using a multi-stage membrane process comprising a combination of low rejection filtration membranes and high rejection filtration membranes employed at a pressure outside of and above the manufacturer's established operating pressure. In certain exemplary embodiments, the improvement in flavor retention is observed when the pressure employed comprises at least about 700 psi.

In an embodiment, when a liquid-based product, for example a 100% juice, is concentrated with a multi-stage membrane process, greater than 30% of the volatile components in the product are retained as compared to the same product prior to concentration. Alternately, at least 50% of the volatile components in the product are retained, or at least 75% of the volatile components in the product are retained, or at
least 85% of the volatile components in the product are retained, or at least 90% of the volatile components in the product are retained, as compared to the same product before undergoing concentration. In contrast, thermal concentration methods often result in the retention of about 15% of volatile components, for instance between 15% and 30% of flavor and aroma compounds in 100% orange juice.

In an embodiment, when a liquid-based food or beverage product, for example and without limitation orange juice, is concentrated with a multi-stage membrane process, the resulting concentrated product is devoid of the compound hydroxymethylfurfural (HMF). In contrast, a disadvantage of thermal concentration methods is that exposure to heat can cause the formation of HMF in the concentrated product through the dehydration of particular sugars, such as fructose. Once formed, HMF remains in the concentrated product, and thus its presence acts as an indicator of exposure to heat. For instance, exposure to temperatures between about 90 and 95 degrees Fahrenheit for a time of about three months has been observed to generate at least one thousand parts per billion (ppb) HMF in food and beverage products, such as at least 2000 ppb HMF. In contrast, fresh juice products typically contain only as much as about 200 to 300 ppb HMF. The amount of HMF in a sample may readily be measured using analytical instruments, for example using high pressure liquid chromatography (HPLC) with ultraviolet (UV) detection. According to at least certain aspects, products concentrated according to multi-stage membrane processes disclosed herein exhibit a HMF content of less than about 500 ppb HMF, or less than about 300 ppb HMF, or less than 200 ppb HMF.

An advantage of at least certain aspects of the present invention comprises an improvement in sensory perception, in particular texture perception, as a result of the filtration processing steps. Without wishing to be bound by theory, it is believed that the particle sizes of particles in the product are decreased due to being subjected to shear while passing through one or more pumps in the multi-stage membrane apparatus. This effect may be measured by determining the particle size distribution in the concentrated product or reconstituted product. In an embodiment, the particle size distribution is affected by the one or more of the filtration steps, resulting in a significant increase in the concentration of particles smaller than approximately 5 microns in the liquid after concentration of the filtration retentate. Such a shift in
particle size distribution results in a smoother texture, which may be a more desirable texture in certain applications.

[68] In embodiments of the invention, a concentrated product is provided comprising a brix of at least 40 degrees brix and at least 75% of the nutrients as the product prior to concentration, the nutrients comprising vitamins, carbohydrates, proteins, fibers and phytonutrients. Optionally, the product comprises less than 500 ppb hydroxymethylfurfural (HMF) and brix is at least 42 degrees brix or at least 60 degrees brix.

[69] As discussed above, higher concentrations of the product are also achievable with the concentration methods disclosed herein, such as at least 35 degrees brix, or at least 42 degrees brix, or at least 65 degrees brix. Similarly, a reconstituted product is achieved using any combination of high rejection and low rejection membranes, comprising a liquid (e.g., water) and the concentrated product, where the product comprises a from concentrate 100% fruit juice or vegetable juice comprising a flavor, an aroma, or combinations thereof, that are statistically equal to or superior to the flavor and/or the aroma of the same product instead comprising a from concentrate fruit or vegetable juice that was thermally concentrated. Similarly, the product optionally comprises a from concentrate 100% fruit juice or vegetable juice comprising a flavor, an aroma, or combinations thereof, that are statistically identical to the flavor and/or the aroma of a not from concentrate fruit or vegetable juice, as determined by a sensory panel. For example, the product optionally comprises a from concentrate 100% orange juice product comprising a flavor and an aroma that are statistically identical to the flavor and the aroma of a not from concentrate orange juice, as determined by a sensor’ panel.

[70] It is an advantage of at least certain embodiments of the invention to provide a concentrated product from which little or none of the nutrients are lost in the filtration permeates during the filtration processes. For example and without limitation, nutrients comprising vitamins, carbohydrates, proteins, fibers and phytonutrients are retained in the filtration retentates when the products are concentrated under a pressure of at least 250 psi, for example at least 700 psi. Liquid-based products for use in embodiments of the present invention and derived from fruits and vegetables advantageously provide macro nutrition, micro nutrition, or combinations thereof, to
finished products according to embodiments of the invention. As used herein, the term "macro nutrition" refers to components that provide nutrients in a relatively large amount, for example and without limitation, protein, carbohydrates, fat, fiber, and combinations thereof. As used herein, the term "micro nutrition" refers to components that provide nutrition in relatively small amounts, for example and without limitation, vitamins, electrolytes, minerals, trace minerals, phytonutrients such as flavonoids, carotenoids, and combinations thereof. For example and without limitation, the solids may provide fiber, vitamins such as vitamin C and vitamin A, flavonoids, carotenoids such as lycopene, and combinations thereof. Accordingly, the macro nutrition, the micro nutrition, or both, of a composition in embodiments of the invention may be manipulated by selecting liquid-based products derived from a specific fruit, vegetable, or combinations thereof. For instance, when it is desired to provide a composition comprising flavonoids, citrus products may be concentrated via the inventive methods.

[71] The type and amount of nutrients in an initial product may be determined using analytical methods known to those of skill in the art, such as gas chromatography - mass spectrometry (GC-MS), high pressure liquid chromatography (HPLC), UV-VIS spectroscopy, and the like. In aspects comprising a reconstituted orange juice, a from concentrate 100% orange juice product is provided comprising an amount of nutrients that is statistically identical to the amount of nutrients in a not from concentrate orange juice. In alternate aspects, a from concentrate 100% fruit or vegetable juice product is provided comprising an amount of nutrients that is within 75% of the amount of nutrients in the same fruit or vegetable juice that is not from concentrate, or within 80% of the amount of nutrients, or within 90% of the amount of nutrients, or within 95% of the amount of nutrients, or within 99% of the amount of nutrients.

[72] In at least certain embodiments of the invention, the separation of solids from the water soluble phase produces a reconstituted product (i.e., comprising a liquid and the concentrated product) comprising measured viscosities that are significantly lower than the starting liquid product. Furthermore, as a filtration permeate is removed from each sequential filtration retentate, the retentate viscosity' does not increase at the same rate as for products that contain suspended material. This phenomenon is believed to aid in the separation process at the operational pressures of about
250 psi and about 3,500 psi: lower viscosities allow higher pressures and higher cross-flow velocities to be applied during the membrane concentration process.

[73] Referring to FIG. 2, a theoretical depiction of the filtration of a liquid through a membrane is provided. FIG. 2 shows the concentration of solutes (i.e., components being concentrated in the liquid-based product) as the bulk solution begins to approach the membrane, and how the flow decreases in the boundary layer. Moreover, FIG. 2 illustrates that the concentration of solutes at the membrane wall is equivalent to the concentration in the gel layer directly adjacent to the membrane wall. The thickness of the gel layer is controlled by adjusting the cross-flow velocity; as discussed above, a decrease in cross-flow velocity increases the thickness of the gel layer whereas an increase in cross-flow velocity decreases the thickness of the gel layer. Last, FIG. 2 depicts the decrease in concentration of the solutes in the membrane permeate that passes through the membrane.

[74] In at least certain embodiments of the invention, a from concentrate reconstituted product is provided comprising a titratable acidity that is less than the titratable acidity of the same not from concentrate product, wherein the concentrated product has not been subjected to ion exchange or chromatographic processes. Titratable acidity is indicative of a solution's total acidity, and is measured by reacting the acids present with a base (e.g., sodium hydroxide) to a selected end point of about pH 7.0. The end point may be determined using a pH meter or via a pH-sensitive color indicator. Without wishing to be bound by theory, it is believed that the filtration processes allow at least some low molecular weight acids to pass through the membrane into the filtration permeate, thereby lowering the amount of acid in each filtration retentate. It is an advantage of certain aspects that the methods do not require an additional ion exchange or other chromatographic process to reduce the acid content of the concentrated product, saving time, expense and materials.

[75] Juice beverages containing 100% juice are popular with consumers for numerous reasons, such as their nutritional profile and lack of added water. These juice beverages must meet particular standard of identity criteria. The US Food and Drug Administration sets a standard for food labeling, including juice labeling. 21 CFR Section 101.30 states that beverages containing "100 percent juice and non-juice ingredients that do not result in a diminution of the juice soluble solids or, in the case
of expressed juice, in a change in the volume, when the 100 percent juice declaration appears on a panel of the label that does not also bear the ingredient statement, it must be accompanied by the phrase "with added ___." the blank filled in with a term such as "ingredient(s)," "preservative," or "sweetener," as appropriate (e.g., "100% juice with added sweetener"), except that when the presence of the non-juice ingredient(s) is declared as a part of the statement of identity of the product, this phrase need not accompany the 100 percent juice declaration." Consequently, since solids derived from a fruit or a vegetable are ingredients included within the standard of identity of juice, the juice beverages of certain embodiments of the invention may be labeled as "100 percent juice."

[76] Similarly, not from concentrate (NFC) juices tend to be popular with consumers for numerous reasons, such as their fresh taste and nutritional profile. These NFC juices also must meet particular standard of identity criteria. Among these criteria are brix minmums and brix-to-acid ratio minimums. For example, the US Food and Drag Administration sets a standard for juices such as orange juice. In this regard 21 CFR Section 146.140, incorporated by reference hereinto, states that finished pasteurized orange juice is to contain not less than 10.5 percent by weight of orange juice soluble solids, exclusive of the solids of any added sweetening ingredients. This FDA regulation further states that the ratio of brix to grams of citric acid per 100 ml of juice is not less than a 10 to 1 ratio. The juice industry recognizes these criteria for pasteurized orange juice or single strength orange juice as applying to NFC orange juice. It will be understood that these standard of identity criteria are used herein with respect to NFC orange juice or pasteurized single strength orange juice. This same concept of standard of identity criteria applies as well to other pasteurized single strength juices. In certain embodiments, the juice beverages according to the current invention meet the criteria of NFC juice.

[77] Liquids derived from one or more fruits, one or more vegetables, and combinations thereof, are a basic ingredient in juice beverages disclosed here, typically being the vehicle or primary liquid portion in which the remaining ingredients are dissolved, emulsified, suspended or dispersed. Liquids suitable for use in at least certain exemplary embodiments of the food and beverage products disclosed here include, e.g., fruit, vegetable and berry juices. Liquids can be employed in the present
invention in the form of a single-strength juice, NFC juice, 100% pure juice, juice concentrate, serum, clarified juice, fruit or vegetable water, clarified serum, or other suitable forms. The term "clarified" as used herein refers to a liquid that has had the solid matter removed using filtration or centrifugation. Typically, the filtration comprises removing solid matter as small as 0.1 microns in diameter. The term "serum" as used herein refers to the thin, clear portion of the fluid of plants, such as fruits or vegetables. The term "water" as used herein refers to the clear liquid extracted from fruits or vegetables. The term "juice" as used herein includes single-strength fruit (including berry) or vegetable juice, as well as concentrates, milks, and other forms. Multiple liquids derived from different fruits and/or vegetables can be combined to generate a juice beverage having the desired nutrients.

Examples of suitable liquid sources include, without limitation, orange, lemon, lime, tangerine, mandarin orange, tangelo, pomelo, grapefruit, grape, red grape, sweet potato, tomato, celery, beet, lettuce, spinach, cabbage, watercress, rhubarb, carrot, cucumber, raisin, cranberry, pineapple, peach, banana, apple, pear, guava, apricot, watermelon, Saskatoon berry, blueberry, plains berry, prairie berry, mulberry, elderberry, Barbados cherry (acerola cherry), choke cherry, date, coconut, olive, raspberry, strawberry, huckleberry, loganberry, currant, dewberry, boysenberry, kiwi, cherry, blackberry, quince, buckthorn, passion fruit, sloe, rowan, gooseberry, pomegranate, persimmon, mango, rhubarb, papaya, lychee, plum, prune, date, currant, fig, etc. Numerous additional and alternative liquids derived from fruits or vegetables suitable for use in at least certain exemplary embodiments will be apparent to those skilled in the art given the benefit of this disclosure.

In alternative embodiments, juice beverages may be prepared that are not 100% juice. For example, juice beverages may comprise from concentrate (FC) juice, which is juice that has been previously concentrated to remove water, and then diluted to provide at least a minimum specified Brix, depending on the type of juice. Orange juice, for instance, must have a minimum Brix level of 11.8, while grapefruit juice must have a minimum Brix level of 10.0. Further embodiments include juice beverages comprising reduced calorie, light, or low-calorie juice. Such beverages typically comprise juice, added water, and often other added ingredients to provide a desired taste, such as non-nutritive sweeteners.
In certain embodiments, both pulp and homogenized finisher-derived solids are incorporated into juice beverages. The inclusion of both pulp and homogenized solids provides a combination of the advantages of both materials, as well as the opportunity to specifically select amounts and varietals of pulp and solids based on the characteristics of the materials.

It should be understood that food and beverage products, such as juice beverages and other juice beverage products, in accordance with this disclosure may have any of numerous different specific formulations or constitutions. In general, a from concentrate 100% juice beverage in accordance with this disclosure typically comprises a liquid and at least one concentrated fruit or vegetable juice. The formulation of a food or beverage product in accordance with this disclosure can vary to a certain extent, depending upon such factors as the product's intended market segment, its desired nutritional characteristics, flavor profile and the like. For example, it will generally be an option to add further ingredients to the formulation of a particular food or beverage embodiment, in particular if the juice beverage is not required to meet a specific standard of identity. Additional (i.e., more and/or other) sweeteners may be added, flavorings, inclusions (e.g., fruit or vegetable pieces, fiber, oat flour or nuts), electrolytes, vitamins, tastants, masking agents and the like, flavor enhancers, and/or carbonation typically can be added to any such formulations to vary the taste, mouthfeel, nutritional characteristics, etc.

In embodiments comprising a from concentrate reconstituted product, water may be the vehicle or primary liquid portion in which the concentrated product and any additional ingredients are included. Purified water can be used in the manufacture of certain embodiments of the food and beverage products disclosed here, and water of a standard quality can be employed in order not to adversely affect food or beverage taste, odor, or appearance. The water typically will be clear, colorless, free from objectionable minerals, tastes and odors, free from organic matter, low in alkalinity and of acceptable microbiological quality based on industry and government standards applicable at the time of producing the beverage. In certain embodiments, water is present at a level of from about 1% to about 99.9% by weight of the food or beverage product. In at least certain exemplary embodiments the water used in products disclosed here is "treated water," which refers to water that has been treated
to reduce the total dissolved solids of the water prior to optional supplementation, e.g., with calcium as disclosed in U.S. Patent No. 7,052,725. Methods of producing treated water are known to those of ordinary skill in the art and include deionization, distillation, filtration and reverse osmosis ("r-o"), among others. The terms "treated water," "purified water," "demineralized water," "distilled water," and "r-o water" are understood to be generally synonymous in this discussion, referring to water from which substantially all mineral content has been removed, typically containing no more than about 500 ppm total dissolved solids, e.g. 250 ppm total dissolved solids.

[83] Acid used in food and beverage products disclosed here can serve any one or more of several functions, including, for example, providing antioxidant activity, lending tartness to the taste of the product, enhancing palatability, increasing thirst quenching effect, modifying sweetness and acting as a mild preservative by providing microbiological stability. Ascorbic acid, commonly referred to as "vitamin C", is often employed as an acidulant in foods and beverages to also provide a vitamin to the consumer. Any suitable edible acid may be used, for example citric acid, malic acid, tartaric acid, phosphoric acid, ascorbic acid, lactic acid, formic acid, fumaric acid, gluconic acid, succinic acid and/or adipic acid.

[84] The acid can be used in solid or solution form, and in an amount sufficient to provide the desired pH of the food or beverage product. Typically, for example, the one or more acids of the acidulant are used in amount, collectively, of from about 0.01% to about 1.0% by weight, e.g., from about 0.05% to about 0.5% by weight, such as 0.1% to 0.25% by weight of the product, depending upon the acidulant used, desired pH, other ingredients used, etc. The amount of acid in the product may range from about 1.0% to about 2.5%, between about 1.5% and about 2.0%, or about 1.8% by weight of the product. In certain embodiments of the invention, all of the acid included in a composition may be provided by citric acid.

[85] The pH of at least certain exemplary embodiments of the food and beverage products disclosed here can be a value within the range of 2.5 to 4.0. The acid in certain exemplary embodiments can enhance product flavor. Too much acid can impair the flavor and result in sourness or other off-taste, while too little acid can make the product taste flat and reduce microbiological safety of the product. It will be within the ability of those skilled in the art, given the benefit of this disclosure, to select a
suitable acid or combination of acids and the amounts of such acids for the acidulant component of any particular embodiment of the food and beverage products disclosed here.

[86] Sweeteners suitable for use in various embodiments of the food and beverage products disclosed here include nutritive and non-nutritive, natural and artificial or synthetic sweeteners. In at least certain exemplary embodiments of the products disclosed here, the sweetener component can include nutritive, natural crystalline or liquid sweeteners such as sucrose, liquid sucrose, fructose, liquid fructose, glucose, liquid glucose, glucose-fructose syrup from natural sources such as apple, chicory, honey, etc., e.g., high fructose corn syrup, invert sugar, maple syrup, maple sugar, honey, brown sugar molasses, e.g., cane molasses, such as first molasses, second molasses, blackstrap molasses, and sugar beet molasses, sorghum syrup, Lo Han Guo juice concentrate and/or others. Typically, such sweeteners are present in a gel beverage concentrate in an amount of from about 0.5% to about 35% by weight, such as from about 15 to about 25% by weight. Further, such sweeteners are present in an amount of from about 0.1% to about 20% by weight of a finished food or beverage, such as from about 6% to about 16% by weight, depending upon the desired level of sweetness for the food or beverage. To achieve desired product uniformity, texture and taste, in certain exemplary embodiments of beverage products disclosed here, standardized liquid sugars as are commonly employed in the beverage industry can be used. Typically such standardized sweeteners are free of traces of nonsugar solids which could adversely affect the flavor, color or consistency of the beverage.

[87] Suitable non-nutritive sweeteners and combinations of sweeteners are selected for the desired nutritional characteristics, taste profile for the product, mouthfeel and other organoleptic factors. Non-nutritive sweeteners suitable for at least certain exemplary embodiments include, but are not limited to, for example, peptide based sweeteners, e.g., aspartame, neotame, and alitame, and non-peptide based sweeteners, for example, sodium saccharin, calcium saccharin, acesulfame potassium, sodium cyclamate, calcium cyclamate, neohesperidin dihydrochalcone, and sucralose. In certain embodiments the sweetener comprises acesulfame potassium. Other non-nutritive sweeteners suitable for at least certain exemplary embodiments include, for example, Stevia rebaudiana extracts, rebaudioside A, rebaudioside B, rebaudioside C,
rebaudioside D, rebaudioside E, rebaudioside F, sorbitol, mannitol, xylitol, glycyrrhizin, D-tagatose, erythritol, meso-erythritol, maltitol, maltose, lactose, fructo-oligosaccharides, Lo Han Guo powder, xylose, arabinose, isomalt, lactitol, maltitol, trehalose, and ribose, and protein sweeteners such as thaumatin, monellin, brazzem, L-alanine and glycine, related compounds, and mixtures of any of them. Lo Han Guo, Stevia rebaudiana extracts, rebaudioside A, and monatin and related compounds are natural non-nutritive potent sweeteners.

Non-nutritive, high potency sweeteners typically are employed at a level of milligrams per fluid ounce of beverage, according to their sweetening power, any applicable regulatory provisions of the country where the beverage is to be marketed, the desired level of sweetness of the beverage, etc. It will be within the ability of those skilled in the art, given the benefit of this disclosure, to select suitable additional or alternative sweeteners for use in various embodiments of the beverage products disclosed here.

Preservatives may be used in certain embodiments of the food and beverage products disclosed here. That is, certain exemplar embodiments contain an optional dissolved preservative system. Solutions with a pH below 4.6 and especially those below 3 typically are "microstable," i.e., they resist growth of microorganisms, and so are suitable for longer term storage prior to consumption without the need for further preservatives. However, an additional preservative system can be used if desired. Furthermore, embodiments of juice beverages having low acidity generally comprise a preservative system. If a preservative system is used, it can be added to the beverage product at any suitable time during production, e.g., in some cases prior to the addition of the sweetener. As used here, the terms "preservation system" or "preservatives" include all suitable preservatives approved for use in food and beverage compositions, including, without limitation, such known chemical preservatives as benzoic acid, benzoates, e.g., sodium, calcium, and potassium benzoate, sorbates, e.g., sodium, calcium, and potassium sorbate, citrates, e.g., sodium citrate and potassium citrate, polyphosphates, e.g., sodium hexametaphosphate (SHMP), lauryl arginate ester, cinnamic acid, e.g., sodium and potassium cinnamates, polylysine, and antimicrobial essential oils, dimethyl dicarbonate, and mixtures
thereof, and antioxidants such as ascorbic acid, EDTA, BHA, BHT, TBHQ, EMIQ, dehydroacetic acid, ethoxyquin, heptylparaben, and combinations thereof.

[90] Preservatives can be used in amounts not exceeding mandated maximum levels under applicable laws and regulations. The level of preservative used typically is adjusted according to the planned final product pH, as well as an evaluation of the microbiological spoilage potential of the particular beverage formulation. The maximum level employed typically is about 0.05% by weight of the beverage. It will be within the ability of those skilled in the art, given the benefit of this disclosure, to select a suitable preservative or combination of preservatives for beverages according to this disclosure. In certain embodiments of the invention, sorbic acid or its salts (sorbates) may be employed as preservatives in the products, such as in an amount of less than 0.1% by weight of a beverage concentrate.

[91] Other methods of food and beverage preservation suitable for at least certain exemplar embodiments of the products disclosed here, such as finished beverages, partially finished beverages, soups, snacks, ready-to-drink beverages, etc., include, e.g., aseptic packaging and/or heat treatment or thermal processing steps, such as hot filling and tunnel pasteurization. Such steps can be used to reduce yeast, mold and microbial growth in the beverage products. For example, U.S. Patent No. 4,830,862 to Braun et al. discloses the use of pasteurization in the production of fruit juice beverages as well as the use of suitable preservatives in carbonated beverages. U.S. Patent No. 4,925,686 to Kastin discloses a heat-pasteurized freezable fruit juice composition which contains sodium benzoate and potassium sorbate. In general, heat treatment includes hot fill methods typically using high temperatures for a short time, e.g., about 190° F for 10 seconds, tunnel pasteurization methods typically using lower temperatures for a longer time, e.g., about 160° F for 10-15 minutes, and retort methods typically using, e.g., about 250° F for 3-5 minutes at elevated pressure, i.e., at pressure above 1 atmosphere.

[92] The products disclosed here optionally contain a flavoring composition, for example, natural and synthetic fruit flavors, botanical flavors, other flavors, and mixtures thereof. As used here, the term "fruit flavor" refers generally to those flavors derived from the edible reproductive part of a seed plant. Included are both those wherein a sweet pulp is associated with the seed, e.g., banana, tomato, cranberry and the like,
and those having a small, fleshy berry. The term berry also is used here to include aggregate fruits, i.e., not "true" berries, but that are commonly accepted as a berry. Also included within the term "fruit flavor" are synthetically prepared flavors made to simulate fruit flavors derived from natural sources. Examples of suitable fruit or berry sources include whole berries or portions thereof, berry juice, berry juice concentrates, berry purees and blends thereof, dried berry powders, dried berry juice powders, and the like.

Exemplary fruit flavors include the citrus flavors, e.g., orange, lemon, lime and grapefruit, and such flavors as apple, pomegranate, grape, cherry, and pineapple flavors and the like, and mixtures thereof. In certain exemplary embodiments beverage concentrates and beverages comprise a fruit flavor component, e.g., a juice concentrate or juice. As used here, the term "botanical flavor" refers to flavors derived from parts of a plant other than the fruit. As such, botanical flavors can include those flavors derived from essential oils and extracts of nuts, bark, roots and leaves. Also included within the term "botanical flavor" are synthetically prepared flavors made to simulate botanical flavors derived from natural sources. Examples of such flavors include cola flavors, tea flavors, and the like, and mixtures thereof. The flavor component can further comprise a blend of the above-mentioned flavors. The particular amount of the flavor component useful for imparting flavor characteristics to the products of the present invention will depend upon the flavor(s) selected, the flavor impression desired, and the form of the flavor component. Those skilled in the art, given the benefit of this disclosure, will be readily able to determine the amount of any particular flavor component(s) used to achieve the desired flavor impression.

Other flavorings suitable for use in at least certain exemplary embodiments of the products disclosed here include, e.g., spice flavorings, such as mint, cassia, clove, cinnamon, pepper, ginger, vanilla spice flavorings, cardamom, coriander, root beer, sassafras, ginseng, and others. Numerous additional and alternative flavorings suitable for use in at least certain exemplary embodiments will be apparent to those skilled in the art given the benefit of this disclosure. Flavorings can be in the form of an extract, oleoresin, juice concentrate, bottler's base, or other forms known in the art. In at least certain exemplary embodiments, such spice or other flavors complement that of a juice or juice combination.
The one or more flavorings can be used in the form of an emulsion. A flavoring emulsion can be prepared by mixing some or all of the flavorings together, optionally together with other ingredients of the beverage, and an emulsifying agent. The emulsifying agent may be added with or after the flavorings mixed together. In certain exemplary embodiments the emulsifying agent is water-soluble. Exemplary suitable emulsifying agents include gum acacia, modified starch, carboxymethylcellulose, gum tragacanth, gum ghatti and other suitable gums. Additional suitable emulsifying agents will be apparent to those skilled in the art of beverage formulations, given the benefit of this disclosure. The emulsifier in exemplary embodiments comprises greater than about 3% of the mixture of flavorings and emulsifier. In certain exemplary embodiments the emulsifier is from about 5% to about 30% of the mixture.

Carbon dioxide can be used to provide effervescence to certain exemplary embodiments of the products disclosed here, such as juice beverages, or frozen slush beverages, for instance. Any of the techniques and carbonating equipment known in the art for carbonating beverages can be employed. Carbon dioxide can enhance the beverage taste and appearance and can aid in safeguarding the beverage purity by inhibiting and destroying objectionable bacteria. In certain embodiments, for example, the beverage has a CO₂ level up to about 7.0 volumes carbon dioxide. Typical embodiments may have, for example, from about 0.5 to 5.0 volumes of carbon dioxide. As used here and independent claims, one volume of carbon dioxide is defined as the amount of carbon dioxide absorbed by any given quantity of water at 60° F (16° C) temperature and atmospheric pressure. A volume of gas occupies the same space as does the water by which it is absorbed. The carbon dioxide content can be selected by those skilled in the art based on the desired level of effervescence and the impact of the carbon dioxide on the taste or mouthfeel of the beverage. The carbonation can be natural or synthetic.

The products disclosed here may contain additional ingredients, including, generally, any of those typically found in food and beverage formulations. Examples of such additional ingredients include, but are not limited to, salt, caffeine, caramel and other coloring agents or dyes, antifoaming agents, gums, emulsifiers, tea solids, cloud components, and mineral and non-mineral nutritional supplements. Examples of non-
mineral nutritional supplement ingredients are known to those of ordinary skill in the art and include, for example, antioxidants and vitamins, including Vitamins A, D, E (tocopherol), C (ascorbic acid), B₁ (thiamine), B₂ (riboflavin), B₃ (nicotinamide), B₄ (adenine), B₅ (pantothenic acid, calcium), B₆ (pyridoxine HCl), B₁₂ (cyanocobalamin), and K₁ (phyloquinone), niacin, folic acid, biotin, and combinations thereof. The optional non-mineral nutritional supplements are typically present in amounts generally accepted under good manufacturing practices. Exemplary amounts are between about 1% and about 100% RDV, where such RDV are established. In certain exemplary embodiments the non-mineral nutritional supplement ingredient(s) are present in an amount of from about 5% to about 20% RDV, where established.

Notwithstanding the claims, the invention is also defined by way of the following clauses:

1. A method for providing liquid beverage or an edible foodstuff comprising the steps of:
   - passing a product through a first filtration membrane thereby providing a first filtration retentate and a first filtration permeate;
   - passing the first filtration permeate through a second filtration membrane thereby providing a second filtration retentate and a second filtration permeate.

2. Method according to clause 1 comprising the further step of passing the second filtration retentate through a third filtration membrane thereby providing a third filtration retentate and a third filtration permeate.

3. Method according to clause 2 further comprising passing the third filtration retentate through a fourth filtration membrane, thereby providing a fourth filtration retentate and a fourth filtration permeate.

4. Method according to clause 3 further comprising passing the fourth filtration retentate through a fifth filtration membrane, thereby providing a fifth filtration retentate and a fifth filtration permeate.

5. Method according to any of the preceding clauses comprising passing the product through a plurality of filtration membranes, and/or further comprising the
steps of passing the fifth filtration retentate through a sixth filtration membrane to provide a sixth filtration retentate which is passed through a seventh filtration membrane.

6. Method according to any of the preceding clauses comprising the further step of passing one or more of the first filtration permeate, the second filtration permeate, the third filtration permeate, the fourth filtration permeate, and the fifth filtration permeate through one or more of the first, second, third, fourth and fifth filtration membranes.

7. Method according to any of the preceding clauses wherein one or more of the first, second, third, fourth and fifth filtration membranes are selected from the group consisting of a reverse osmosis membrane, a nanofiltration membrane, an ultrafiltration membrane, a hollow fiber membrane, a tubular membrane, a stainless steel membrane, a ceramic membrane, a spiral wound membrane.

8. Method according to any of the preceding clauses wherein the first filtration membrane having a molecular weight cutoff (MWCO) greater than 500,000 Daltons.

9. Method according to any of the preceding clauses wherein the third filtration membrane has a MWCO of between 1,000 and 10,000 Daltons.

10. Method according to any of the preceding clauses wherein the first filtration permeate is passed through the second filtration membrane using a pressure of between 250 psi and 3,500 psi, preferably of between 700 psi and 2,200 psi and/or wherein the second filtration permeate is passed through the third filtration membrane using a pressure of between 250 psi and 3,500 psi, or at least 700 psi, preferably of between 700 psi and 2,200 psi, and/or wherein the pressure is above and outside of the membrane manufacturer specification, and/or wherein the third filtration permeate is passed through the fourth filtration membrane using a pressure of between 250 psi and 3,500 psi, preferably of between 700 psi and 2,200 psi, and/or wherein the fourth filtration permeate is passed through the fifth filtration membrane using a pressure of between 250 psi and 3,500 psi, preferably of between 700 psi and 2,200 psi, and/or
wherein any of the membranes are used at a pressure outside and above the membrane manufacturer specification.

11. Method according to clause 10 wherein the second, third, fourth and fifth filtration membranes have an effective sugar rejection capability of between about 10% and 99.9 % sugar rejection, for example of between about 10% and 90 % sugar rejection, preferably of between about 90% and about 99.9% sugar rejection.

12. Method according to any of the preceding clauses wherein any of the first filtration retentate, the second filtration retentate, the third filtration retentate, the fourth filtration retentate, and the fifth filtration retentate, are combined together to form a concentrated product.

13. The method according to any of the preceding clauses wherein the concentrated product comprises a brix of greater than 35 degrees brix, preferably wherein the concentrated product comprises a brix of greater than 42 degrees brix, more preferably wherein the concentrated product comprises a brix of greater than 65 degrees brix.

14. The method according to any of the preceding clauses wherein each of the filtration membranes comprises a material independently selected from an inorganic material or a polymeric material.

15. The method according to any of the preceding clauses wherein the product is selected from the group consisting of one or more fruit juices, one or more vegetable juices, a beverage product, a soup, and a liquid snack.

16. The method according to any of the preceding clauses further comprising pasteurizing and/or clarifying the product prior to passing the product through the first filtration membrane, and/or pasteurizing and/or clarifying the concentrated product.

17. The method according to any of the preceding clauses wherein the concentrated product comprises less than 500 ppb hydroxymethylfurfural (HMF).
18. The method according to any of the preceding clauses wherein the product is selected from the group consisting of one or more fruit juices, one or more vegetable juices, a beverage product, a soup, and a liquid snack.

19. The method according to any of the preceding clauses further comprising diluting the concentrated product with water to form a 100% orange juice product comprising a flavor and an aroma that are statistically equal to or superior to the flavor and the aroma of a from concentrate orange juice comprising the same brix and in which the orange juice was thermally concentrated, as determined by a sensor’s panel.

20. The method according to any of the preceding clauses for separating a component from a liquid-based composition comprising passing a liquid-based composition that does not contain insoluble solids through the first filtration membrane wherein the component comprises a molecular weight at least ten times smaller than the molecular weight cut-off of the second filtration membrane.

21. The method according to any of the preceding clauses comprising:

passing a clarified product through a plurality of filtration membranes independently selected from the group consisting of a hollow fiber membrane, a tubular membrane, a spiral wound membrane, a stainless steel membrane, and a ceramic membrane, thereby providing at least one filtration retentate comprising a brix value of at least 65 brix and at least one filtration permeate; wherein at least one of the plurality of filtration membranes comprises an ultrafiltration membrane or a nanofiltration membrane used at a pressure above and outside of the manufacturer specification.

22. A concentrated product obtainable according to a method of any of the preceding clauses.

23. A concentrated product comprising:

a brix of at least 40 degrees brix; and

at least 75% of the nutrients as the product prior to concentration, the nutrients comprising vitamins, carbohydrates, proteins, fibers and phytonutrients.
24. Concentrated product of clause 22 or 23, wherein the product comprises less than 500 ppb hydroxymemylfurfural (HMF).

25. Concentrated product of any of the clauses 22-24 wherein the brix is at least 65 degrees brix.

26. A product comprising water and the concentrated product of any of the clauses 22-25 wherein the product comprises a flavor and an aroma that are statistically equal to or superior to the flavor and the aroma of the same from concentrate product in which the product was thermally concentrated, as determined by a sensory panel.

27. Product of clause 27, wherein the product comprises an amount of nutrients within 80% of the amount of nutrients of the same product prior to concentration.

28. Product according to clause 26 or 27 being beverage, snack or soup.

29. The product according to any of the clauses 26 or 27 comprising a 100% orange juice product comprising a flavor and an aroma that are statistically identical to the flavor and the aroma of a not from concentrate orange juice, as determined by a sensory panel.

30. The product of clause 29, wherein the 100% orange juice product comprises an amount of nutrients that is within 80% of the amount of nutrients in a not from concentrate orange juice.

31. The product according to any of the clauses 22-27 comprising a from concentrate 100% orange juice product comprising a flavor and an aroma that are statistically identical to the flavor and the aroma of a not from concentrate orange juice, as determined by a sensory panel.

32. The product of clause 29, wherein the 100% orange juice product comprises an amount of nutrients that is within 80% of the amount of nutrients in a not from concentrate orange juice.

33. The product of any of the preceding clauses 22-27 wherein the product comprises a from concentrate 100% juice product comprising a flavor and an aroma
that are statistically identical to the flavor and the aroma of a not from concentrate
juice, as determined by a sensory panel.

34. Product comprising water and the concentrated product of clause 33, wherein
the product comprises a from concentrate 100% juice product comprising at least 75%
of the volatile compounds of the same juice prior to concentration, preferably wherein
the product comprises at least 90% of the volatile compounds of the same juice prior
to concentration, more preferably

35. Product according to any of the preceding clauses 22-27, 29-34 comprising a
100% orange juice product.

36. A system of filtration membranes for use in the method of any of the clauses
1-21, for producing a product according to any of the clauses 22-35.

EXAMPLES

Example 1

The capability of a membrane to effectively filter a sugar solution when operated at
various pressures was tested. A sugar solution containing water, high fructose corn
syrup and sucrose, was prepared to simulate a forty brix orange juice, the solution
containing ratios of sugars that mimic the ratios of sucrose to fructose to glucose of 2 : 1 : 1 found in a typical orange juice.

The 40 brix simulated juice was passed through a commercially available polyamide
spiral wound nanofiltration membrane, which has a salt rejection of between about
90% and about 99.9% magnesium sulfate. The simulated juice was passed through
the membrane at approximately 50 degrees Fahrenheit (10 degrees Celsius) at
pressures ranging from about 90 psi to about 1,400 psi and at a cross-flow velocity
between about 41 and 46 centimeters per second (cm/s). The membrane has a
manufacturer's recommended operating pressure of up to about 100 psi. Three
replicates were tested, and the percent rejection of sugars was measured at each
pressure. The resulting measured percent rejection values are shown in the graph of
FIG. 3. As shown in FIG. 3, the percent rejection of sugars increased as pressure
increased, including up to the highest tested pressure of about 1,400 psi. The brix of each permeate was measured, and decreased as the pressure increased. For instance, the permeate brix at a filtration pressure of about 90 psi was approximately 35 brix, the permeate brix at a filtration pressure of about 1,000 psi was approximately 17 brix, and the permeate brix at a filtration pressure of about 1,400 psi was approximately 14 brix. Accordingly, even starting with a high brix solution of 40 brix, it is possible to achieve significant separation of a sugar solution at filtration pressures well above and outside the membrane manufacturer's established operating pressure range.

Example 2

[101] The capability of another membrane to effectively filter a sugar solution when operated at various pressures was tested. A sugar solution containing water, high fructose corn syrup and sucrose, was prepared to simulate a forty brix orange juice, the solution containing ratios of sugars that mimic the ratios of sucrose to fructose to glucose of 2 : 1 : 1 found in a typical orange juice.

[102] The 40 brix simulated juice was passed through a commercially available polyethersulfone (PES) spiral wound membrane having a molecular weight cutoff (MWCO) of about 5,000 Daltons, which has a salt rejection of between about 90% and about 99.9% magnesium sulfate. The simulated juice was passed through the membrane at approximately 51 degrees Fahrenheit (10.6 degrees Celsius) at pressures ranging from about 45 psi to about 2,200 psi and at a cross-flow velocity between about 41 and 46 cm/s. The membrane has a manufacturer's recommended operating pressure of up to about 100 psi. Three replicates were tested, and the percent rejection of sugars was measured at each pressure. The resulting measured percent rejection values are shown in the graph of FIG. 4. As shown in FIG. 4, the percent-rejection of sugars increased as pressure increased, including up to the highest tested pressure of about 2,200 psi. The brix of each permeate was measured, and decreased as the pressure increased. For instance, the permeate brix at a filtration pressure of about 45 psi was approximately 35 brix, the permeate brix at a filtration pressure of about 1,000 psi was approximately 28 brix, and the permeate brix at a filtration pressure of about 2,200 psi was approximately 20 brix. Accordingly, even starting with a high brix solution of 40 brix, it is possible to achieve significant separation of a
sugar solution at filtration pressures well above and outside the membrane manufacturer's established operating pressure range.

Example 3

[103] The capability of a membrane to effectively filter a sugar solution when operated at various pressures was tested. A sugar solution containing water, high fmctose corn syrup and sucrose, was prepared to simulate a fifty brix orange juice, the solution containing ratios of sugars that mimic the ratios of sucrose to fmctose to glucose of 2 : 1 : 1 found in a typical orange juice.

[104] The 50 brix simulated juice was passed through a commercially available polyamide spiral wound nanofiltration membrane, which has a salt rejection of between about 90% and about 99.9% magnesium sulfate. The simulated juice was passed through the membrane at approximately 50 degrees Fahrenheit (10 degrees Celsius) at pressures ranging from about 400 psi to about 1,600 psi and at a cross-flow velocity between about 41 and 46 cm/s. The membrane has a manufacturer's recommended operating pressure of up to about 100 psi. Three replicates were tested, and the percent rejection of sugars was measured at each pressure. The resulting average measured percent rejection values are shown in the graph of FIG. 5. As shown in FIG. 5, the percent rejection of sugars increased as pressure increased, including up to the highest tested pressure of about 1,600 psi. The brix of each permeate was measured, and decreased as the pressure increased. For instance, the permeate brix at a filtration pressure of about 400 psi was approximately 41 brix, the permeate brix at a filtration pressure of about 800 psi was approximately 36 brix, and the permeate brix at a filtration pressure of about 1,600 psi was approximately 26 brix. Accordingly, even starting with a high brix solution of 50 brix, it is possible to achieve significant separation of a sugar solution at filtration pressures well above and outside the membrane manufacturer's established operating pressure range.

Example 4

[105] The capability of another membrane to effectively filter a sugar solution when operated at various pressures was tested. A sugar solution containing water, high fmctose corn syrup and sucrose, was prepared to simulate a fifty brix orange juice, the
solution containing ratios of sugars that mimic the ratios of sucrose to fructose to glucose of 2 : 1 : 1 found in a typical orange juice.

[106] The 50 brix simulated juice was passed through a commercially available polyethersulfone (PES) spiral wound membrane having a molecular weight cutoff (MWCO) of about 5,000 Daltons, which has a salt rejection of between about 90% and about 99.9% magnesium sulfate. The simulated juice was passed through the membrane at approximately 50 degrees Fahrenheit (10 degrees Celsius) at pressures ranging from about 400 psi to about 2,200 psi and at a cross-flow velocity between about 41 and 46 cm/s. The membrane has a manufacturer's recommended operating pressure of up to about 100 psi. Three replicates were tested, and the percent rejection of sugars was measured at each pressure. The resulting average measured percent rejection values are shown in the graph of FIG. 6. As shown in FIG. 6, the percent rejection of sugars increased as pressure increased, including up to the highest tested pressure of about 2,200 psi. The brix of each permeate was measured, and decreased as the pressure increased. For instance, the permeate brix at a filtration pressure of about 400 psi was approximately 45 brix, the permeate brix at a filtration pressure of about 800 psi was approximately 42 brix, and the permeate brix at a filtration pressure of about 2,200 psi was approximately 32 brix. Accordingly, even starting with a high brix solution of 50 brix, it is possible to achieve significant separation of a sugar solution at filtration pressures well above and outside the membrane manufacturer's established operating pressure range.

Example 5

[107] The capability of another membrane to effectively filter a sugar solution when operated at various pressures was tested. A sugar solution containing water, high fructose corn syrup and sucrose, was prepared to simulate a sixty brix orange juice, the solution containing ratios of sugars that mimic the ratios of sucrose to fructose to glucose of 2 : 1 : 1 found in a typical orange juice.

[108] The 60 brix simulated juice was passed through a commercially available polyethersulfone (PES) spiral wound membrane having a molecular weight cutoff (MWCO) of about 5,000 Daltons, which has a salt rejection of between about 90% and about 99.9% magnesium sulfate. The simulated juice was passed through the membrane at approximately 50 degrees Fahrenheit (10 degrees Celsius) at pressures
ranging from about 550 psi to about 2,200 psi and at a cross-flow velocity between about 41 and 46 cm/s. The membrane has a manufacturer's recommended operating pressure of up to about 100 psi. Three replicates were tested, and the percent rejection of sugars was measured at each pressure. The resulting average measured percent rejection values are shown in the graph of FIG. 7. As shown in FIG. 7, the percent rejection of sugars increased as pressure increased, including up to the highest tested pressure of about 2,200 psi. The brix of each permeate was measured, and decreased as the pressure increased. For instance, the permeate brix at a filtration pressure of about 550 psi was approximately 57 brix, the permeate brix at a filtration pressure of about 1,400 psi was approximately 54 brix, and the permeate brix at a filtration pressure of about 2,200 psi was approximately 48 brix. Accordingly, even starting with a high brix solution of 60 brix, it is possible to achieve significant separation of a sugar solution at filtration pressures well above and outside the membrane manufacturer's established operating pressure range.

Example 6

[109] The discovery of the capability of significantly increasing the rejection of a membrane to concentrate a solution can also be extended to the general principle that applying a pressure above and outside the manufacturer's established operating pressure will selectively separate specific components from a composition. This is evidenced by each of FIGS 3-6, which shows the correlation between pressure and percent rejection. Accordingly, particular components can be targeted for separation by choosing the appropriate filtration pressure.

[110] Moreover, it was discovered that aspects of the present invention selectively separate disaccharides from monosaccharides. Specifically, FIG. 8 is a graph showing the difference in the content of sucrose (i.e., a disaccharide) and the content of glucose (i.e., a monosaccharide) in simulated juice solutions. The solutions had a brix of either 30 degrees brix, 40 degrees brix, or 50 degrees brix, and were passed through a commercially available polyamide spiral wound nanofiltration membrane, which has a salt rejection of between about 90% and about 99.9% magnesium sulfate, at approximately 69.8 degrees Fahrenheit (21 degrees Celsius) at a pressure of 800 psi and at a cross-flow velocity of about 60 cm/s. The glucose and sucrose contents of each permeate was measured, and the results are plotted in FIG. 8. It can be seen that
although each solution feed comprises an equal amount of glucose and sucrose, sucrose was preferentially retained during filtration through the membrane. In particular, the filtration permeates contained between about 3 to about 3.7 times as much glucose as sucrose.

Example 7

[111] A sample of not from concentrate orange juice comprising a brix often degrees, from which solids greater than 500,000 Daltons had been removed, was concentrated according to an embodiment of the invention. The ten degree brix orange juice was passed through a high rejection reverse osmosis membrane comprising an effective sugar rejection of between about 90% and about 99.9% sugar rejection at twenty degrees Celsius and at a pressure of 829 psi. The ultrafiltration retentate comprised a concentration of about 31 degrees brix.

[112] The 31 brix retentate was next passed through a second high rejection reverse osmosis membrane comprising an effective sugar rejection of between about 90% and about 99.9% sugar rejection at twenty degrees Celsius and at a pressure of 1790 psi. The second reverse osmosis retentate comprised a concentration of about 46 degrees brix.

[113] The 46 brix retentate was then passed through a low rejection ultrafiltration membrane comprising an effective sugar rejection of between about 10% and about 75% sugar rejection at twenty degrees Celsius and at a pressure of 745 psi. The second ultrafiltration retentate comprised a concentration of about 60 degrees brix. Consequently, it is possible to achieve significant concentration of an orange juice serum at filtration pressures well above and outside the membrane manufacturer’s established operating pressure range.

Example 8

[114] The capability of a membrane to effectively reject small, water-soluble volatile compounds present in a sugar solution when operated at various pressures was tested. A sugar solution containing water, high fructose corn syrup and sucrose, was prepared to simulate a 37.5 brix orange juice, the solution ratios of sugars that mimic the ratios of sucrose to fructose to glucose of 2:1:1 found in a typical orange juice. In addition, an aliquot of small, water-soluble volatile compounds including aldehydes,
esters, alcohols, terpene hydrocarbons, ketones, and compounds associated with off-notes, which are typically found in orange juice, were added to the sugar solution. Such compounds are often added to juice, as a so-called "add back" to replace small volatile compounds that can be lost during processing, storage, or combinations thereof, of the juice.

The 37.5 brix simulated juice was passed through a commercially available polyethersulfone (PES) spiral wound membrane having a molecular weight cutoff (MWCO) of about 5,000 Daltons, which has a salt rejection of between about 90% and about 99.9% magnesium sulfate. The simulated juice was passed through the membrane at approximately 50 degrees Fahrenheit (10 degrees Celsius) at pressures ranging from about 50 psi to about 2,200 psi and at a cross-flow velocity of about 45 cm/s. The membrane has a manufacturer's recommended operating pressure of up to about 100 psi. Three replicates were tested, and the percent rejection of compounds was measured at each pressure.

The resulting measured percent rejection values for one alcohol compound, iinalool, which was included in the simulated juice in an amount of 1.04 mg/L (ppm), are shown in the graph of FIG. 9. The chemical name for iinalool is 3,7-dimethylocta-1,6-dien-3-ol, and it has a molecular weight of 154.25 g/mol and a vapor pressure of 0.0905 millimeters of mercury (mm/Hg) at twenty-five degrees Celsius. As shown in FIG. 9, the percent rejection of iinalool generally increased as pressure increased, including up to the highest tested pressure of about 2,200 psi. At a pressure of 100 psi, within the manufacturer's specifications, the amount of iinalool rejected was 16.3%. In contrast, at a pressure of 1,400 psi, the amount of iinalool rejected was 43.3%. Accordingly, it was a surprising discovery that it is possible to achieve significant retention of a small, water-soluble and volatile compound in a sugar solution at filtration pressures well above and outside the membrane manufacturer's established operating pressure range.

Moreover, the brix of each permeate was measured, and decreased as the pressure increased. For instance, the permeate brix at a filtration pressure of about 50 psi was approximately 31 brix, the permeate brix at a filtration pressure of about 1,000 psi was approximately 28 brix, and the permeate brix at a filtration pressure of about 2,200 psi was approximately 20 brix.
Given the benefit of the above disclosure and description of exemplary embodiments, it will be apparent to those skilled in the art that numerous alternate and different embodiments are possible in keeping with the general principles of the invention disclosed here. Those skilled in this art will recognize that all such various modifications and alternative embodiments are within the true scope and spirit of the invention. The appended claims are intended to cover all such modifications and alternative embodiments. It should be understood that the use of a singular indefinite or definite article (e.g., "a," "an," "the," etc.) in this disclosure and in the following claims follows the traditional approach in patents of meaning "at least one" unless in a particular instance it is clear from context that the term is intended in that particular instance to mean specifically one and only one. Likewise, the term "comprising" is open ended, not excluding additional items, features, components, etc.
We claim:

1. A method comprising:
   passing a product through a first filtration membrane selected from the group consisting of a hollow fiber membrane, a tubular membrane, a stainless steel membrane, and a ceramic membrane, the first filtration membrane having a molecular weight cutoff (MWCO) greater than 500,000 Daltons, thereby providing a first filtration retentate and a first filtration permeate;
   passing the first filtration permeate through a second filtration membrane using a pressure of between 250 psi and 3,500 psi, thereby providing a second filtration retentate and a second filtration permeate; and
   passing the second filtration retentate through a third filtration membrane using a pressure above and outside of the membrane manufacturer specification comprising between 250 psi and 3,500 psi, thereby providing a third filtration retentate and a third filtration permeate.

2. The method of claim 1, further comprising combining the first and third filtration retentates to form a concentrated product.

3. The method of claim 1, wherein the second filtration membrane has an effective sugar rejection capability of between about 90% and about 99.9% sugar rejection.

4. The method of claim 3, wherein the first filtration permeate is passed through the second filtration membrane at a pressure between 700 psi and 2,200 psi.

5. The method of claim 3, wherein the third filtration membrane has an effective sugar rejection capability of between about 10% and about 90% sugar rejection.

6. The method of claim 1, wherein the second filtration membrane has an effective sugar rejection capability of between about 10% and about 99% sugar rejection.

7. The method of claim 6, wherein the third filtration membrane has an effective sugar rejection capability of between about 90% and about 99.9% sugar rejection.
8. The method of claim 2, wherein the concentrated product comprises a brix of greater than 35 degrees brix.

9. The method of claim 8, wherein the concentrated product comprises a brix of greater than 42 degrees brix.

10. The method of claim 9, wherein the concentrated product comprises a brix of greater than 65 degrees brix.

11. The method of claim 1, further comprising passing the second filtration retentate through a third filtration membrane, the third filtration membrane selected from an ultrafiltration membrane and a nanofiltration membrane, thereby providing a third filtration retentate and a third filtration permeate.

12. The method of claim 11, further comprising passing the third filtration retentate through a fourth filtration membrane, the third filtration membrane selected from an ultrafiltration membrane and a nanofiltration membrane, thereby providing a fourth filtration retentate and a fourth filtration permeate.

13. The method of claim 12, further comprising passing the fourth filtration retentate through a fifth filtration membrane, thereby providing a fifth filtration retentate and a fifth filtration permeate.

14. The method of claim 11, wherein the concentrated product comprises a brix of at least 65 degrees brix.

15. The method of claim 12, further comprising combining each of the first and fourth filtration retentates to form a concentrated product.

16. The method of claim 13, further comprising combining each of the first and fifth filtration retentates to form a concentrated product.
17. The method of claim 1, wherein each of the first filtration membrane and the second filtration membrane comprises a material independently selected from an inorganic material or a polymeric material.

18. The method of claim 1, wherein the first filtration permeate is passed through the second filtration membrane using a pressure of at least 700 psi and no more than 2,200 psi.

19. The method of claim 1, wherein the product is selected from the group consisting of one or more fruit juices, one or more vegetable juices, a beverage product, a soup, and a liquid snack.

20. The method of claim 1, further comprising pasteurizing the product prior to passing the product through the first filtration membrane.

21. The method of claim 1, further comprising passing the second filtration permeate and the third filtration permeate through the second filtration membrane.

22. The method of claim 14, further comprising pasteurizing the concentrated product.

23. The method of claim 15, wherein the concentrated product comprises less than 500 ppb hydroxymethylfurfural (HMF).


25. A concentrated product comprising:
   a brix of at least 40 degrees brix; and
   at least 75% of the nutrients as the product prior to concentration, the nutrients comprising vitamins, carbohydrates, proteins, fibers and phytonutrients.

26. The concentrated product of claim 25, wherein the product comprises less than 500 ppb hydroxymethylfurfural (HMF).

27. The concentrated product of claim 26, wherein the brix is at least 65 degrees brix.
28. A product comprising water and the concentrated product of claim 25, wherein the product comprises a flavor and an aroma that are statistically equal to or superior to the flavor and the aroma of the same from concentrate product in which the product was thermally concentrated, as determined by a sensory panel.

29. The product of claim 28, wherein the product comprises an amount of nutrients within 80% of the amount of nutrients of the same product prior to concentration.

30. A product comprising water and the concentrated product of claim 26, wherein the product comprises a 100% orange juice product comprising a flavor and an aroma that are statistically identical to the flavor and the aroma of a not from concentrate orange juice, as determined by a sensory panel.

31. The product of claim 30, wherein the 100% orange juice product comprises an amount of nutrients that is within 80% of the amount of nutrients in a not from concentrate orange juice.

32. A product comprising water and the concentrated product of claim 27, wherein the product comprises a from concentrate 100% orange juice product comprising a flavor and an aroma that are statistically identical to the flavor and the aroma of a not from concentrate orange juice, as determined by a sensory panel.

33. The product of claim 28, wherein the 100% orange juice product comprises an amount of nutrients that is within 80% of the amount of nutrients in a not from concentrate orange juice.

34. The product of claim 27, wherein the product comprises a from concentrate 100% juice product comprising a flavor and an aroma that are statistically identical to the flavor and the aroma of a not from concentrate juice, as determined by a sensory panel.

35. A product comprising water and the concentrated product of claim 27, wherein the product comprises a from concentrate 100% juice product comprising at least 75% of the volatile compounds of the same juice prior to concentration.
36. The product of claim 35, wherein the product comprises at least 90% of the volatile compounds of the same juice prior to concentration.

37. The product of claim 35, wherein the product comprises a 100% orange juice product.

38. A method comprising passing a clarified product through a first filtration membrane, the first filtration membrane selected from a reverse osmosis membrane, a nanofiltration membrane and an ultrafiltration membrane, thereby providing a first filtration retentate and a first filtration permeate;

   passing the first filtration retentate through a second filtration membrane having an effective sugar rejection capability of between about 10% and 99.9% sugar rejection using a pressure of between 250 psi and 3,500 psi, thereby providing a second filtration retentate and a second filtration permeate;

   passing the second filtration retentate through a third filtration membrane, the first filtration membrane selected from a nanofiltration membrane and an ultrafiltration membrane, using a pressure of between 250 psi and 3,500 psi, thereby providing a third filtration retentate and a third filtration permeate; and

   passing each of the first filtration permeate, the second filtration permeate and the third filtration permeate through the first filtration membrane.

39. The method of claim 38, further comprising passing the third filtration retentate through a fourth filtration membrane, thereby providing a fourth filtration retentate and a fourth filtration permeate.

40. The method of claim 38, further comprising combining each of the first and third filtration retentates to form a concentrated product.

41. The method of claim 39, further comprising combining each of the first and fourth filtration retentates to form a concentrated product.

42. The method of claim 39, wherein the third filtration retentate is passed through the fourth filtration membrane using a pressure of at least 700 psi and no more than 2,200 psi.
43. The method of claim 38, wherein the product is selected from the group consisting of one or more fruit juices, one or more vegetable juices, a beverage product, a soup, and a liquid snack.

44. The method of claim 38, further comprising diluting the concentrated product with water to form a 100% orange juice product comprising a flavor and an aroma that are statistically equal to or superior to the flavor and the aroma of a from concentrate orange juice comprising the same brix and in which the orange juice was thermally concentrated, as determined by a sensor's panel.

45. A method of selectively separating a component from a liquid-based composition comprising:
   passing a liquid-based composition that does not contain insoluble solids through a filtration membrane using a pressure of between 250 psi and 3,500 psi, thereby providing a first filtration retentate and a first filtration permeate; and
   passing the first filtration retentate through a second filtration membrane using a pressure of between 250 psi and 3,500 psi, thereby providing a second filtration retentate and a second filtration permeate, wherein the component comprises a molecular weight at least ten times smaller than the molecular weight cut-off of the second filtration membrane.

46. A product comprising water and a concentrated product, wherein the concentrated product comprises a juice product comprising a brix of at least 65 degrees brix, wherein the product comprises a 100% juice product comprising a flavor and an aroma that are statistically equal to or superior to the flavor and the aroma of the same juice prepared by combining water and a concentrated juice comprising the same brix and concentrated using thermal methods, as determined by a sensory panel.

47. A method comprising:
   passing a product through a first filtration membrane selected from the group consisting of a hollow fiber membrane, a tubular membrane, a stainless steel membrane, and a ceramic membrane, the first filtration membrane having a molecular weight cutoff (MWCO) greater than 500,000 Daltons, thereby providing a first filtration retentate and a first filtration permeate;
passing the first filtration permeate through a second filtration membrane using a pressure above and outside of the membrane manufacturer specification, thereby providing a second filtration retentate and a second filtration permeate; and

passing the second filtration retentate through a third filtration membrane using a pressure above and outside of the membrane manufacturer specification, thereby providing a third filtration retentate and a third filtration permeate.

48. The method of claim 47, wherein the first filtration permeate is passed through the second filtration membrane at a pressure of between 250 psi and 3,500 psi.

49. The method of claim 48, wherein the second filtration membrane has an effective sugar rejection capability of between about 10% and about 99.9% sugar rejection.

50. The method of claim 47, wherein the second filtration membrane is selected from the group consisting of a spiral wound membrane, a tubular membrane, a hollow fiber membrane, a flat sheet plate and frame membrane, and a ceramic membrane.

51. A method comprising:

passing a product through a first filtration membrane selected from the group consisting of a hollow fiber membrane, a tubular membrane, a stainless steel membrane, and a ceramic membrane, the first filtration membrane having a molecular weight cutoff (MWCO) greater than 500,000 Daltons, thereby providing a first filtration retentate and a first filtration permeate;

passing the first filtration permeate through a second filtration membrane, thereby providing a second filtration retentate comprising a brix value of at least 35 degrees brix and a second filtration permeate; and

passing the second filtration retentate through a third filtration membrane, the third filtration membrane selected from an ultrafiltration membrane and a nanofiltration membrane, thereby providing a third filtration retentate comprising a brix value of at least 40 degrees brix and a third filtration permeate.

52. A concentrated product made by the method of claim 51.
53. The method of claim 51, wherein the second filtration retentate is passed through the third filtration membrane using a pressure of at least 700 psi.

54. The method of claim 51, further comprising passing the third filtration retentate through a fourth filtration membrane, the fourth filtration membrane selected from an ultrafiltration membrane and a nanofiltration membrane, thereby providing a fourth filtration retentate and a fourth filtration permeate.

55. The method of claim 54, wherein the fourth filtration retentate comprises a brix value of at least 65 degrees brix.

56. A method comprising:
   passing a product through a first filtration membrane selected from the group consisting of a hollow fiber membrane, a tubular membrane, a stainless steel membrane, and a ceramic membrane, the first filtration membrane having a molecular weight cutoff (MWCO) greater than 500,000 Daltons, thereby providing a first filtration retentate and a first filtration permeate;
   passing the first filtration permeate through a second filtration membrane, thereby providing a second filtration retentate comprising a brix value of at least 35 brix and a second filtration permeate; and
   passing the second filtration retentate through a third filtration membrane, the third filtration membrane comprising a MWCO of between 1,000 and 10,000 Daltons, thereby providing a third filtration retentate and a third filtration permeate.

57. A method comprising:
   passing a clarified product through a plurality of filtration membranes independently selected from the group consisting of a hollow fiber membrane, a tubular membrane, a spiral wound membrane, a stainless steel membrane, and a ceramic membrane, thereby providing at least one filtration retentate comprising a brix value of at least 65 brix and at least one filtration permeate; wherein at least one of the plurality of filtration membranes comprises an ultrafiltration membrane or a nanofiltration membrane used at a pressure above and outside of the manufacturer specification.
Fig. 5

REJECTION AT 50 BRIX

TMP (PSI)

50 45 40 35 30 25 20 15 10 5

(%) REJECTION
Fig. 8
SUGAR RATIO OF FEED AND PERMEATE PER CONCENTRATION

- GLUCOSE
- SUCROSE

CONCENTRATION g/L

30 BRIX FEED
30 BRIX PERMEATE
40 BRIX FEED
40 BRIX PERMEATE
50 BRIX FEED
50 BRIX PERMEATE
INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2013/027273

A. CLASSIFICATION OF SUBJECT MATTER
IPC(8) - A23L 2/74 (2013.01)
USPC - 426/495
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED
Minimum documentation searched (classification system followed by classification symbols)
IPC(8) - A23L 2/72, 2/74; B01D 61/58 (2013.01)
USPC - 210/295, 314, 323.1, 323.2, 335, 908; 426/330.5; 490, 495, 616

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
CPC - A23L 2/72, 2/74; B01D 61/58 (2013.01)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
Orbit, Google Patents, Google Scholar, Google

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1-24, 38-44, 47-57</td>
</tr>
<tr>
<td>Y</td>
<td>US 6,440,222 B1 (DONOVAN et al) 27 August 2002 (27.08.2002) entire document</td>
<td>1-24, 47-57</td>
</tr>
</tbody>
</table>

Further documents are listed in the continuation of Box C.

* = Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"Q" document member of the same patent family

Date of the actual completion of the international search
05 June 2013

Date of mailing of the international search report
19 JUN 2013

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Form PCT/ISA/210 (second sheet) (July 2009)
# INTERNATIONAL SEARCH REPORT

## Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. [ ] Claims Nos.: because they relate to subject matter not required to be searched by this Authority, namely:

2. [ ] Claims Nos.: because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. [ ] Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

See Extra Sheet

1. [ ] As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. [ ] As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.

3. D As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. [X] No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.: 1-24, 38-45, 47-57

### Remark on Protest

- [ ] The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- [ ] The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- [ ] No protest accompanied the payment of additional search fees.

Form PCT/ISA/210 (continuation of first sheet (2)) (July 2009)
This application contains the following inventions or groups of inventions which are not so linked as to form a single general inventive concept under PCT Rule 13.1. In order for all inventions to be examined, the appropriate additional examination fees must be paid.

Group I, claims 1-24, 38-45, and 47-57, are drawn to a filtration method.

Group II, claims 25-37 and 46, are drawn to a concentrated product.

The inventions listed as Groups I and II do not relate to a single general inventive concept under PCT Rule 13.1 because, under PCT Rule 13.2, they lack the same or corresponding special technical features for the following reasons:

The special technical features of the Group I, a product treatment method comprising passing a product through first, second, and third filtration membranes under pressure, are not present in Group II; and the special technical features of the Group II, a concentrated product having a specified degrees Brix and nutrients, are not present in Group I.

Groups I and II share the technical features of a concentrated product having a specified degrees Brix and nutrients formed by filtration having a flavor determined by a sensory panel. However, these technical features do not represent a contribution over the prior art.

Specifically, US 2011/0305816 A1 to Dubbelman et al. discloses a concentrated product (Para. [0008] applicants have found a more cost effective process to prepare an umami active tomato fraction which can be used to add and/or augment the umami taste in food products; Para. [0011] ...concentrating the at least one first portion low in lycopene) having a specified degrees Brix and nutrients (Para. [0015] ...concentrating said second fraction, preferably to a Brix value of at least 10...whereby said second fraction is used as the umami active tomato fraction) formed by filtration (Para. [0037] and [0077] regarding filtration processes) having a flavor determined by a sensory panel (Para. [0085] regarding impact on flavor of the umami active fraction obtained, sensory evaluations have been performed by a trained sensory panel).

Since none of the special technical features of the Groups I and II inventions are found in more than one of the inventions, unity of invention is lacking.