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(54) Title: OSMOTIC CONCENTRATION (57) Abstract There is disclosed an osmotic concentration cell wherein the flow chamber of the osmotic concentration cell has a continually changing flow path to provide a region of high turbulence to a semipermeable membrane defining the flow chamber. High turbulence at a first semipermeable membrane interface significantly reduces membrane fouling during osmotic concentration and allows for concentration cell to be used for a variety of purposes including, for example, producing low alcohol wine, concentrating a thin wine into a premium wine, concentrating a low quality grape juice into a higher quality grape juice for fermenting a more premium wine, concentrating various fruit or vegetable juices, and concentrating various beverages, such as teas or coffee.		

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OSMOTIC CONCENTRATION

TECHNICAL FIELD OF THE INVENTION

The present invention relates to an osmotic concentration apparatus and osmotic
5 concentration cell having a configuration which promotes turbulence at a first membrane surface
to prevent fouling of the membrane surface. The cell may also be used as a dialysis cell. The
present invention further relates to a method for making a low alcohol wine or other fermented
beverage, a method for improving the quality of a wine beverage, a process to provide a grape
juice concentrate from mid to early harvest grapes that can be used for a desert wine, and a
10 method for improving the quality of a grape juice concentrate used for wine fermentation.

BACKGROUND OF THE INVENTION

Osmotic concentration cells are often used to concentrate food products, such as fruit
juices, by selectively removing water. In osmotic concentration cells, water diffuses from the
15 food product to be concentrated through a semipermeable membrane into a solution having a
much lower water content. Osmotic concentration systems are of interest to concentrate food
products without use of heat that may damage the food product, such as a fruit juice or a grape
juice used in wine making. This prevents much of the quality degradation associated with
evaporative processes. Food products of particular interest for osmotic concentration include
20 other fruit and vegetable juices, agricultural products such as aloe vera, and pharmaceutical and
biopharmaceutical products.

Most juice concentrates are made using evaporators. This is the source of quality
problems with concentrates due to loss of volatile fragrances, denaturation and self-oxidation of
the product. Volatiles are lost during the evaporative process because volatile agents have a
25 lower boiling point than water. Some of the volatiles can be recovered by distillation of the
vapor, however this system is, at best, only partially successful and it adds tremendous costs to
the overall process. Loss of volatiles degrades product quality because the volatile species are
responsible for the fragrance associated with various products, such as fresh juice. Self
oxidation is primarily a problem for juices. The term refers to enzymatic destruction of colors
30 and flavors in the juice by naturally-occurring enzymes. The enzymes are derived from the juice
and are largely inactive at refrigeration temperatures. However, such enzymes can become
active at the higher temperatures associated with commercial evaporation.

Previous attempts to solve the problems of loss of volatiles and self-oxidation during
concentration of juices include various cold concentration techniques. For food products, cold
35 techniques include freeze concentration, reverse osmosis (RO) and direct osmotic concentration
(DOC). Freeze concentration can eliminate self-oxidation problems and produce a higher
quality juice than evaporators. However there are problems associated with freeze
concentration, including: (1) only clarified juices can be concentrated, (2) some minor juice
constituents are lost, and (3) capital costs of a commercial scale freeze concentration plant is

many times that of an evaporation plant. Accordingly, the costs for freeze concentration are prohibitive, making this process not economically feasible, especially in wine making operations.

RO techniques have lower capital and operating costs than freeze concentration.

However, RO capital costs still far exceed evaporation techniques capital and operating costs.

5 RO operates by squeezing water out of a juice through a semipermeable membrane (a thin polymer which has been manufactured to be porous). In semipermeable membranes, the pore structure is small enough that small molecules, such as water, can pass through, while larger organic molecules are prevented from passing. Drawbacks to RO are that the membranes foul rapidly and extremely high pressures (*e.g.*, 3000 psi) are needed to form a juice concentrate.

10 Direct osmotic concentration (DOC) has been studied less than other cold concentration processes. DOC uses semipermeable membranes. However, instead of squeezing water out with pressure, DOC uses a solution with a lower mole fraction of water to pull water out of a product. This solution with a low mole fraction of water is an osmotic agent (OA). In DOC, any water pulled from the product into the OA must be subsequently removed from the OA,
15 preferably by evaporation, in order for the OA to be recycled. Evaporation of the OA does not effect product quality because the product itself is not heated. Since DOC requires an evaporation step, its energy requirement is similar to evaporative concentration.

Concentrating products containing large organic molecules and solutions with suspended solids (*e.g.*, pulp from orange juice) creates severe membrane fouling problems, particularly in
20 RO systems. Even when a product is first filtered to remove suspended solids, there is severe fouling due to deposition of films on the surfaces of the semipermeable membrane. Such films are composed of large molecules such as polypeptides, pectins and polysaccharides. Large organic molecules adhere to the surface of the membrane due to Van der Waals attraction. After attachment, the large molecules slowly denature and unfold and then form multiple
25 hydrogen bonding and multiple ionic interactions with the membrane. Therefore, it is desirable to induce a high degree of turbulence at the membrane surface to sweep these molecules away from the membrane surface before they adhere due to stronger bond formation.

Film breakup in RO systems has been attempted in, for example, Cross, "Membrane Concentration of Orange Juice" *Proc. Fla. State Hort. Soc.* 102, 1989. If the velocity is kept
30 above 1 m/sec in tubular RO systems, enough turbulence is produced that many unfiltered juices can be concentrated up to 30% dissolved solids (30 Brix). Above 30 Brix, turbulence can no longer pull attached molecules away from membrane pores because pressures needed to push water out of the juice at Brix levels above 30 (*i.e.*, greater than 5 MPa) produce pressure gradients at surfaces of membranes greater than 1000 GPa/m. Tubular RO is the RO
35 configuration most resistant to fouling, but requires the greatest amount of space. Plate and frame configurations have greater surface fouling, but are more economical systems to build. Hollow fiber configurations are most susceptible to fouling.

Another method for concentrating juices to a high Brix level in an RO system first removes all large molecules by ultrafiltration before RO concentration. This method allows for

concentration of a 60 Brix juice. However this method is relatively expensive, is difficult to maintain in view of the ultrafiltration step, and is energy intensive.

Flux of water in both RO and DOC systems is controlled by both pressure and osmotic concentration gradients. This can be quantified as:

5
$$F_w = k_w[(P^I - q^I) - (P^{II} - q^{II})]$$

wherein F_w is the water flux, k_w is a constant for flux through a particular membrane, P is pressure, q is the osmotic potential and the I and II superscripts refer to solutions on each side of the membrane. Osmotic potential is a measure of the tendency of water to diffuse through a membrane from a region of high water concentration to a region of lower water concentration.

10 In general, a good approximation of osmotic potential is $q = cRT$, wherein c is the molar concentration of non-water species in a solution, R is a gas constant, and T is the absolute temperature.

In RO systems, pressure differences are large but the osmotic potential of a permeate (the solution water is pushed into) is essentially zero. In DOC systems, pressure differences are negligible and flux depends on the difference in osmotic potential. In RO systems, the value of the flux constant k_w is determined by molecular-scale hydrodynamic resistance to flow through the membrane pores. To minimize this resistance, RO systems use extremely thin membranes (e.g., less than 0.01 mm thick) supported by a porous backing wherein the backing provides structural properties to the membrane to be able to withstand high applied pressure. The pores in the back are much larger than pores in the membrane, hence the backing contributes little to flow resistance across the membrane.

DOC k_w values are primarily controlled by diffusion rates. Diffusion rates are less dependent upon pore diameter than hydrodynamic flow rates. Thus, in DOC cells, the presence of a membrane backing can severely reduce flux rates. Therefore, DOC membranes and their backings, if any, must be as thin as possible.

Further resistance to water flux in DOC systems occurs due to boundary layers on the surfaces of membranes. Boundary layers are thin regions in the fluid next to a membrane surface where mixing with bulk fluid is ineffective. An impact of boundary layers is creation of an extra region that water must diffuse through. Boundary layers are often avoided by using high fluid velocity (turbulent flow if possible) to sweep away boundary layers. Therefore, it is important in this art to design a flow cell configuration to avoid dead spots in fluid flow near membranes. The present invention was made, in part, to solve some of the problems of DOC systems for juice concentrators and dialysis systems.

In the wine industry, there is a need to provide a low alcohol wine to have an alcohol content below 10% to satisfy certain governmental regulations and to satisfy a consumer demand for a wine beverage with full bodied taste and body with less alcohol. There is also a need in the wine industry to improve thin wine fermentation that sometime result and produce a low grade wine. If a thin, low grade wine could be concentrated in both its water and alcohol contents, then it could be transformed into a premium wine and command a higher market price.

There is also a need to improve the quality of various grape juices used for wine fermentation to use a lower grade juice to make a high quality wine. For example the 1992 grape harvest in Italy (a wet season) produced a thin (*i.e.*, low Brix) grape juice and this lowered the quality of the resulting wine. The use of the inventive device according to the inventive process can
5 satisfy these wine industry needs.

There are a number of processes for reducing the alcohol content of fermented drinks, such as wine, beer and champagne. One technique subjects the fermented drink to fractional distillation (usually under a vacuum) in order to remove some ethanol. Fractional distillation usually removes some of the taste and aroma substances from the drink and usually requires
10 addition back to the drink of taste and aroma substances removed. In spite of extraordinary efforts involved in fractional vacuum distillation and rapid evaporators (*e.g.*, thin film evaporators) this process produces poor quality products because the product often does not have the same or nearly the same taste as the original product. In another process, ethanol in a fermented drink may be extracted by means of organic solvents. However, use of organic
15 solvents in this manner would violate many food processing regulations and the taste of the drink is often severely affected. It is known that the flavorings and aroma agents that form the bouquet and other such substances of a wine beverage include about 400 to 600 different substances, such as higher alcohols, esters, aldehydes, ketones, lactones, etc. The total volume of such substances is only about 0.5% to about 1% of the wine volume. However many of
20 these substances are volatile with their boiling points being below and above the boiling point for ethanol. These substances also overlap the corresponding properties of ethanol with regard to their extraction behavior.

Most recent efforts for producing low alcohol beverages have focused upon a dialysis technique to lower alcohol content across a membrane. For example, in U.S. Patent 4,804,554,
25 a hydrophillic membrane is used and the osmotic agent (OA) or "dialysate" is composed of fractionally distilled beverage without alcohol or flavor substances. After each use, the dialysate is "stripped" of alcohol (and other flavoring agents) by fractional distillation. In U.S. Patent 4,581,236 the dialysate is again the alcohol-free beverage. In U.S. Patent 4,664,918, the dialysate is the alcohol-free beverage or an alcohol-free fruit beverage. Use of a dealcoholized
30 product as the OA, however, tends to dilute the product being dialyzed. This happens because removal of alcohol from the juice to be used as the OA lowers the osmotic potential of the solution, with the result that when the OA is introduced into the membrane cell, water is pulled from it into the product being dealcoholized.

Dilution of fermented beverages during alcohol removal is undesirable because, by itself,
35 removal of alcohol decreases the sensory impact of beverage flavors. A dilution of the product further decreases product flavor.

In another technique, U.S. Patent 4,867,997 describes a complicated high-pressure extraction process in the presence of carbon dioxide in a supercritical fluid state. Therefore, there is a need in the art to find an improved process to make a low alcohol fermented beverage

(e.g., wine, beer or champagne) that retains the flavor and aroma characteristics of the original beverage. The present invention was made to achieve such a goal.

SUMMARY OF THE INVENTION

5 The present invention provides an osmotic concentration cell and an osmotic concentration apparatus (comprising an array or plurality of osmotic concentration cells), wherein each cell has a first chamber with a fluid flow design with high turbulence at a first semipermeable membrane surface wherein a flow path of the first chamber has a continual change in flow direction. The osmotic agent (OA) flows on the second side of the
10 semipermeable membrane in a second chamber, preferably in a torturous path between support members, and in the same net direction as the flow and with a slightly lower pressure than the juice.

 In order to accomplish this result, each osmotic concentration cell is designed comprising an enclosed apparatus having one or two semipermeable membranes per cell, each
15 semipermeable membrane having a first side defining a first chamber and a second side defining a second chamber. The semipermeable membrane rests on support members (such as rod-shaped support members) spaced from about 1 cm to about 10 cm apart, wherein the support members are within the second chamber. There is a pressure difference between the juice or first chamber and the OA or second chamber wherein the pressure is greater in the first
20 chamber. This pressure differential deflects the membrane between the support members into the second chamber so that a flow path with a continually changing direction is formed. This flow path is called "corrugated." The juice flow path is continually changing, in a "serpentine" pattern, wherein the radius of curvature of the juice flow path is from about 1.5 cm to about 4.5 cm depending upon the distance between the support members. Preferably, the radius of
25 curvature is about 2.0 cm.

 The osmotic concentration apparatus further comprises one or a plurality of enclosed cells having a flow inlet opening leading to the first chamber of each cell, a flow outlet communicating with the first chamber of each cell, an OA inlet and an OA outlet connected to each second chamber. The second chamber of an osmotic concentration apparatus is defined by
30 internal walls of the osmotic concentration apparatus and the second sides of the semipermeable membranes.

 The present invention further comprises a DOC method for selectively removing water from a juice in an osmotic concentration cell or apparatus, comprising: (a) providing a juice in a continuous flow stream to a juice inlet, (b) pumping the juice at a pressure of from about 1 to
35 about 30 psi through a first chamber and out a juice outlet, and wherein the first chamber comprises a continually changing direction flow path, wherein the radius of curvature of the continually changing direction flow path is from about 1.5 cm to about 4.5 cm., (c) providing an OA in a second chamber, wherein the second chamber is defined by the second side of a semipermeable membrane and enclosed by the cell, and wherein the OA comprises the salt

components of the juice at a molar concentration at least three times and preferably at least five times greater than the undiluted juice. Preferably, the OA further comprises at least 50 weight % of a sugar, wherein the sugar is selected from the group consisting of sucrose, fructose, maltose, glucose, and combinations thereof.

5 The present invention further comprises a method for removing water from grape juice used for wine making, comprising: (a) providing a grape juice to a juice inlet in an osmotic concentration apparatus described herein; (b) providing an OA to the OA chamber, wherein the OA comprises from about 30% to about 85% by weight sugar; (c) applying greater pressure to the grape juice than to the OA to deflect the semipermeable membrane into a corrugated flow
10 path; and (d) removing water from the grape juice. Water is preferably removed from grape juice to produce a grape juice similar to a late-harvest grape juice by increasing the solids content of the grape juice, or to compensate for a short growing season to increase the solids content of the grape juice to make a stronger wine that could not otherwise be produced by nature, or to produce a dessert wine by concentrating grape juice.

15 The present invention further comprises a method for producing a low alcohol wine, comprising: (a) providing wine to a juice inlet in an osmotic concentration apparatus described herein; (b) providing an OA to the OA chamber, wherein the OA comprises from about 10% to about 30% by weight sugar; (c) applying greater pressure to the wine than to the OA to deflect the semipermeable membrane into a corrugated flow path; and (d) removing mostly alcohol
20 from the wine. Preferably, the application of pressure in step (c) is accomplished with nitrogen pressurization to avoid any unnecessary shear pressure on the wine.

 The present invention further comprises a method for strengthening wine, comprising: (a) providing wine to a juice inlet in an osmotic concentration apparatus described herein; (b) providing an OA to the OA chamber, wherein the OA comprises from about 25% to about 50%
25 by weight sugar; (c) applying greater pressure to the wine than to the OA to deflect the semipermeable membrane into a corrugated flow path; and (d) removing approximately equal percentages of alcohol and water from the wine. The inventive method further comprises recycling the OA by selectively distilling off alcohol and water to restore the original weight percentage of sugar in the OA. Preferably, the OA further comprises at least 1 mM of an
30 organic acid. The organic acids in the OA are selected from the group consisting of malic acid, citric acid, tartaric acid, ascorbic acid, and combinations thereof.

BRIEF DESCRIPTION OF THE DRAWINGS

35 Figure 1 illustrates a side view of a portion of an osmotic concentration cell. The supports members are straight bars arranged in parallel which extend into and out of the page. In this cell, the distance between support member centers is about 2.5 cm and the height of the support members is about 2 cm. In operation, the pressure difference between the juice and OA is held below 30 psi and the radius of curvature (*i.e.*, deflection) of the membrane between the support members is about 2.0 cm. The flow path is shown by the curving arrow between the

membranes. Due to a continual change in flow direction, the flow path induces a high degree of turbulence. The OA flows through a gap between the support members (*i.e.*, second chamber), preferably in a direction perpendicular to the juice flow direction.

Figure 2 illustrates a diagram of half (bottom portion) of an inventive DOC apparatus.

5 The top portion of the apparatus (not shown) is identical except for positioning of support members, juice inlet and exit ports. The apparatus shown in Figure 2 is a miniature lab scale pilot model having a top cell and a bottom cell but not having any middle cells. A commercial scale apparatus would have much larger width and length and a greater number of cells (*e.g.* middle cells), however, the spacing between support members and operating pressures would be
10 similar to the illustrated lab scale model.

Figure 3 is a schematic diagram of the inventive direct osmotic concentration (DOC) process to concentrate a juice and remove water from the OA by a evaporation process.

Figure 4 shows a graph of water flux rate from orange juice during a concentration process described in Example 2 herein. A least squares analysis shows that the rate of
15 concentration was linear with time indicating that there was no significant membrane fouling that could impede performance of the apparatus.

Figure 5 shows a graph of flux constant versus time of concentration measuring either the tomato juice or the OA. The line for flux of water into the OA is relatively linear over time, indicating that there is very little, if any, membrane fouling.

20 Figure 6 shows flux from concentrating coffee described in Example 4 herein. The Brix numbers for coffee increased linearly.

Figure 7 shows a stacking design to stack a plurality of modules in a relatively small area.

25 **DETAILED DESCRIPTION OF THE INVENTION**

Device

The present invention provides an osmotic concentration cell and apparatus that have a novel design for flow of a juice providing for high turbulence at a first side of a semipermeable membrane that defines a first chamber. The flow path of the juice to be concentrated is called
30 "corrugated" because there is a continual change in flow path direction. It is this corrugated flow path that provides for a high amount of turbulence at the first side of the membrane and acts to significantly prevent fouling of the membrane. Moreover, presence of suspended solids within the juice can further act as a abrasive agent to continually cleanse the first side of the semipermeable membrane and further prevent membrane fouling.

35 The present invention provides an osmotic concentration cell and an osmotic concentration apparatus (comprising an array or plurality of osmotic concentration cells), wherein each cell has first chamber with a fluid flow design with high turbulence at a first semipermeable membrane surface by having a flow path of the first chamber with a continual

change in flow direction. The osmotic agent (OA) flows on the second side of the semipermeable membrane in a second chamber with a slightly lower pressure than the juice.

The juice, as used herein, refers to any liquid that is to be concentrated by selectively removing water or another species, such as alcohol to create a concentrated juice. In the case of an alcoholic beverage such as wine, the concentrated juice may be a low alcohol wine or a concentrated thin wine. Examples of juices that are concentrated to remove water as the solvent include any fruit juice (*e.g.*, orange, apple, red raspberry, pear, cranberry, guava, grapefruit, lemon, other citrus fruits, tropical fruits or berries), vegetable juices such as tomato or carrot juices, coffees and teas, milk and dairy products and specialty juices such as aloe vera, and perfumes and flavoring agents. Further, other juices may be processed to remove other solvents, such as alcohol and/or water from alcoholic beverages, such as beer or wine to create low alcohol beers or wines or premium wines from thin, low-grade wines. Concentration of a juice is determined by the Brix number. Brix is a measure of percentage of dissolved solids. Therefore, selective removal of a solvent from the juice will increase the percentage of dissolved solids and the Brix number.

In order to selectively remove a particular solvent, each osmotic concentration cell is designed comprising an enclosed apparatus having one or two semipermeable membranes per cell, each semipermeable membrane having a first side defining a first chamber and a second side defining a second chamber. The semipermeable membrane wraps around support members (such as rod-shaped support members) spaced from about 1 cm to about 10 cm apart, wherein the support members are within the second chamber. Preferably, each cell further comprises a sealing device, such as an O-ring that surrounds the semipermeable membrane to seal it and prevent leakage of liquid contents around the semipermeable membrane. The apparatus and cell is made from a rigid material suitable to contact for the particular juice and OA that flows within the cell or apparatus. If food juices are being concentrated, examples of suitable rigid materials include stainless steel, polycarbonate, or other food-grade polymeric materials. The juice preferably contacts only the first surface of the semipermeable membrane and not the material that forms the walls of the cell or apparatus.

The semipermeable membrane is either hydrophobic for non-aqueous juices or hydrophilic for aqueous juices. Examples of hydrophilic membranes include membranes composed of cellulose acetate, cellulose triacetate, polysulfones, other polymeric hydrophilic materials, and composite membranes formed from combinations of the foregoing materials.

There is a pressure difference between the juice or first chamber and the OA or second chamber wherein the pressure is greater in the first chamber to provide membrane deflection within the second chamber between the support members to provide a first chamber juice flow path with a continually changing direction. The maximum pressure difference between the first chamber and the second chamber varies in inverse proportion to the distance between the support members. This flow path is called "corrugated." The juice flow path is continually changing, in a serpentine pattern, wherein the radius of curvature of the juice flow path is from

about 1.5 cm to about 4.5 cm depending upon the distance between the support members. Preferably, the radius of curvature is about 2.0 cm. For example, when there is a 2.5 cm. or 1 inch spacing between support members, appropriate pressure differences between the first and second chambers is from about 1 to about 30 psi.

5 The osmotic concentration apparatus further comprises one or a plurality of enclosed cells having a flow inlet opening leading to the first chamber of each cell, a flow outlet communicating with the first chamber of each cell, an OA inlet and an OA outlet connected to each second chamber. The second chamber of an osmotic concentration apparatus is defined by internal walls of the osmotic concentration apparatus and the second sides of the semipermeable
10 membranes. Preferably, the apparatus is composed of a plurality of stackable module cells. The top and bottom cells comprise the outer walls of the apparatus and one or a plurality of middle cells comprise the middle modules. The middle modules, or cells have no outer walls, but instead have a frame support holding the parallel support members. The semipermeable membranes are placed inside the rows of support members and sealed into place with an
15 appropriate sealing device, such as an O-ring or with an adhesive agent. The semipermeable membranes can be replaced when the modular apparatus is disassembled into its component modules. Each module can be stacked to reduce total area of the apparatus. An example of one stacking arrangement of three modules is shown in Figure 7.

20 The apparatus can be operated in a horizontal, inclined, or preferably, in a vertical orientation. The advantage of vertical orientation is easier removal of bubbles as well as easier assembly and maintenance.

25 The support members are arranged in parallel to support formation of a "corrugated" flow path with each first chamber when greater pressure is applied to the first chamber to deflect the semipermeable membrane into the second chamber. The support members can be any shape so long as there is a rounded surface contacting the second side of the semipermeable
30 membrane. Examples of appropriate shapes are rods, rounded cones or boxes with at least one rounded face.

30 A novel feature of the inventive osmotic concentrator cell is controlled deflection of the semipermeable membrane between support members to provide a flow path with continually changing direction and a high amount of turbulence on the first side of the membrane. This provides for an osmotic concentration cell that can concentrate liquids with gel forming dissolved solids, such as aloe vera or orange juice with pulp that could not otherwise be concentrated in DOC or RO systems.

35 A diagram of a section of a cell is provided in Figure 1. The product being concentrated flows along the first side of a semipermeable membrane that is deflected between two sets of support members by virtue of a higher pressure in the first chamber than in the second chamber. Due to the offset positioning of each set of parallel support members, a corrugated flow pattern is formed in the first chamber. The continuous direction changes for the flow produces a high amount of turbulence at the first side of the semipermeable membrane, the side most prone to

fouling. To improve reliability and allow the use of even thinner membranes, a sheer fabric support can be used as a backing on the second side of the membrane. This support can be either attached to the frame or fused with the membrane.

In addition to high turbulence, the inventive design also reduces membrane stress and susceptibility to bursting. This is important because thin membranes are preferred for DOC systems. According to US Patent 3,228,877, resistance of a membrane to pressure induced bursting is inversely related to the membrane radius of curvature. Therefore a deflection to form a radius of curvature from about 1 to about 4 cm, and preferably from about 2 to about 3 cm, allows the inventive osmotic concentrator cell and apparatus to use extremely thin membranes.

The design of the osmotic concentration cells allows for construction of an osmotic concentration apparatus in a plate and frame type structure. A plate and frame structure allow for the use of relatively inexpensive sheet semipermeable membranes and allows for rapid instillation and replacement of membranes. Such a design is shown in Figure 7. A diagram of the flow paths of an osmotic concentration apparatus is shown in Figure 2. The OA flows in second chambers in a torturous path between support members in the same net direction as the flow. Preferably, membrane seals to prevent leakage between the flow chambers and the OA chambers are made by compressing ends of membranes between gaskets (*i.e.*, O-rings) mounted to boxes in the OA chambers.

Osmotic Agent

The osmotic agent (OA) is designed to osmotically pull the selected solvent (usually water for fruit or vegetable juices or ethanol for beer or wine) from the juice into the OA. The osmotic pressure is initially greater for solutes (*e.g.*, salts, sugars, glycols, and organic volatile agents such as flavors or perfume ingredients) in the OA. The composition of the OA depends upon the particular juice to be concentrated and what are the solutes in solution in the juice. For example, the OA for fruit or vegetable juices can comprise sugars, such as high fructose corn syrup. The question of whether the sugars from the OA migrate into the concentrated juice was investigated by monitoring the stable carbon isotope ratios in the juice and the OA. The ratios of the OA sugar and juice sugar naturally differ to a great extent. It was found that the ratios did not change during the inventive juice concentrating process. Therefore, sugars did not cross the membrane. In the case of lowering the alcohol (ethanol) content of a delicate beverage such as a wine, it is important to not remove flavoring agents. In premium wines, flavoring agents include organic acids such as malic, citric and tartaric acids. The OA should contain less than about 75 weight % sugar.

The OA should be recycled by evaporating the selected solvent removed from the juice. Preferably, from about 1% to about 3% citric acid or ascorbic acid (by weight) is added to the OA to prevent caramelization of any sugars present in the OA during evaporation. The OA only is heated, thus allowing cold concentration of a juice to prevent breakdown of any heat sensitive

components in a juice. The result of the Inventive DOC process using the inventive cell or apparatus is a higher quality concentrated juice that retains its flavor characteristics better than a similar juice concentrated by an evaporative process. Further, the high turbulence at the first side of the semipermeable membrane prevents membrane fouling and leads to longer lasting
5 membranes with more infrequent membrane changes to provide a more cost effective and higher quality juice concentration process.

Juice Concentration Method

The present invention further comprises a DOC method for removing a selected solvent
10 from a juice in an osmotic concentration cell or apparatus, comprising: (a) providing a juice in a continuous flow stream to a juice inlet, (b) pumping the juice at a pressure of from about 1 to about 30 psi through a first chamber and out a juice outlet, wherein the first chamber comprises a continually changing direction flow path, wherein the radius of curvature of the continually
15 changing direction flow path is from about 1.5 cm to about 4.5 cm., (c) providing an OA in a second chamber, wherein the second chamber is defined by the second side of a semipermeable membrane and enclosed by the cell, and wherein the OA comprises the salt components of the juice at a molar concentration at least three times and preferably at least five times greater than the undiluted juice. Preferably, the OA further comprises at least 50 weight % of a sugar,
20 wherein the sugar is selected from the group consisting of sucrose, fructose, maltose, glucose, and combinations thereof.

Wine or Alcoholic Beverage Concentration Methods

The present invention further provides a process for improving the quality of a fruit juice used to make a wine, comprising: (a) providing a thin, low grade fruit juice composition to a
25 juice inlet in an osmotic concentration apparatus described herein; (b) providing an OA to the OA chamber, wherein the OA comprises from about 50% to about 75% by weight sugar; (c) applying greater pressure to the fruit juice than to the OA to deflect the semipermeable membrane into a corrugated flow path; (d) removing water from the fruit juice; and (e) collecting an improved fruit juice product. The inventive method further comprises recycling
30 the OA by selectively distilling off water to restore the original weight percentage of sugar in the OA. Preferably, the OA further comprises at least 1 mM of an organic acid. The organic acids in the OA are selected from the group consisting of malic acid, citric acid, tartaric acid, and combinations thereof.

The present invention further provides a process to make a grape juice that can be used
35 for a desert wine by concentrating the grape juice to about 36 to about 40 degree Brix from a grape juice from "fully ripe" grapes (*i.e.*, approximately 23 degrees Brix) by a process comprising: (a) providing a grape juice composition from fully ripe grapes to a juice inlet in an osmotic concentration apparatus described herein; (b) providing an OA to the OA chamber, wherein the OA comprises from about 50% to about 75% by weight sugar; (c) applying greater

pressure to the grape juice than to the OA to deflect the semipermeable membrane into a corrugated flow path; (d) removing water from the grape juice; and (e) collecting an concentrated grape juice product that can be used to make a desert wine. The inventive method further comprises recycling the OA by selectively distilling off water to restore the original weight percentage of sugar in the OA. Preferably, the OA further comprises at least 1 mM of an organic acid. The organic acids in the OA are selected from the group consisting of malic acid, citric acid, tartaric acid, and combinations thereof.

In general, it is possible to adjust the relative percentages of removal of water and alcohol from a fermented beverage (*i.e.*, wine, beer or champagne) by adjusting the amount of sugar in the OA. Higher amounts of sugar (*i.e.*, from about 30% to about 85%) in the OA removes water faster than alcohol, which can increase the alcohol content of the beverage/wine. Lower sugar concentrations (*i.e.*, from about 3% to about 20%) in the OA removes alcohol faster than water creating a low-alcohol beverage. A beverage/wine can be strengthened without changing the percentage alcohol content by using an intermediate (15-50% brix) concentration OA that removes water and alcohol at rates proportional to their concentrations in the beverage. Therefore, the inventive methods are based upon the discovery that it is not necessary to provide an OA consisting of an alcohol-free beverage or a fruit juice. Instead, the OA need only comprise sugar and water with the amount of sugar controlling the relative rates of water and alcohol removal.

It is further important, when working with a delicate beverage such as a wine, to avoid shearing the beverage. Therefore, the apparatus is preferably pressurized by applying an inert gas (*i.e.*, nitrogen) or at least a gas that avoids oxidation of the beverage, on the juice/wine side of the outlet to maintain the beverage in an inert atmosphere throughout the concentration procedure.

One surprising feature of the inventive apparatus is an ability to concentrate a thin, low grade wine into a wine of much higher quality by removing both alcohol and water from the beverage. When wine is upgraded in this manner, the relative percentage of alcohol within the beverage is usually constant. Therefore, the inventive method using the inventive apparatus can improve wine quality by upgrading lower grade wines (by virtue of a thin body and concentration of favoring agents) into a higher quality wine. This feature is a great significance to the wine industry.

It has been found that a much superior product is made when the product is concentrated slightly (about 5% to about 20%). To accomplish this, a dealcoholized OA with a higher osmotic potential than the full-alcohol beverage must be used (*e.g.*, a sugar solution).

The degree of concentration the product will experience can be adjusted by selecting an appropriate osmotic strength (Brix) of the OA. After processing, the alcohol can be removed from the OA by distillation. Flavors permeating the membrane into the OA can be stripped off before alcohol removal and added back to the OA before the next cycle in order to bring them

into balance with the product. In most cases, however, this is unnecessary. Concentration of the product more than compensates for any flavors lost through the membrane.

EXAMPLE 1

5 This example illustrates the construction of a pilot scale prototype of the inventive DOC apparatus comprising one cell consisting of a top module and a bottom module that was made for conducting the following experiments to concentrate different juices. Additionally, middle modules to create a plurality of cells in an apparatus was made to increase the efficiency of the apparatus by creating additional first chambers to increase the throughput of the DOC
10 apparatus.

 The top module and bottom module are mirror image components (except for offset placement of the support members, *i.e.*, rods) in a rectangular shape. The pilot model was made of polycarbonate in a 28 cm by 100 cm rectangular dimension. Each component is 2.5 cm thick and the interior space houses the parallel rod-shaped support members, also made of
15 polycarbonate affixed by solvent fusing. The support membranes were placed 2.5 cm apart. The interior space is rectangular-shaped and encircled by an O-ring to help seal the membrane. A cellulose triacetate semipermeable membrane having a 100 molecular weight cutoff was placed on each interior, rectangular space such that it slightly overlaps the interior space and is sealed together to form the first chamber when the top and bottom modules are joined to form a
20 DOC cell having one first chamber and two second chambers.

 A middle module was made comprising a polycarbonate rectangular frame of the same dimensions as the top and bottom modules. The interior space of the middle module comprises a row of parallel support members having curved surfaces extending from either side of the middle module. The support members were placed 2.5 cm apart. Further there is a sealing
25 means (*i.e.*, O-rings) surrounding the interior space on both sides of the middle module. Two semipermeable membranes are placed on either side of the interior space of the middle module when the apparatus is assembled. The result is a stackable apparatus with a top, bottom and middle modules having two first chambers and three second chambers. Additional middle modules will increase the number of first chambers and second chambers by one.

30 The clear plastic (polycarbonate) cells allows for visualization of juice and OA flows and determination if there are difficulties with bubbles and the like. Also, the entire apparatus can be moved to a temperature-controlled room, such as a cold room to help to preserve the juice from heat degradation or microbial contamination. Alternatively, the apparatus can be jacketed to control temperatures within the apparatus by pumping an appropriate temperature liquid
35 through the jacket.

EXAMPLE 2

 This example illustrates concentration of an orange juice using the inventive pilot scale apparatus described in Example 1. Approximately 6 liters of fresh orange juice, Brix 12.6, was

obtained. Approximately 6 gallons of OA was prepared with 100% PEG (polyethylene glycol) MW 300. The module had a length of 61 inches, 100 molecular weight cutoff triacetate membrane, and 90 mm membrane thickness.

Figure 4 shows a graph of flux time versus Brix of the orange juice. The orange juice was concentrated to about 50 Brix by transferring water from the orange juice to the OA. The rate of concentration was about linear with time indicating that there was not a significant membrane fouling problem that could impede performance of the apparatus. The Brix of the OA decreased correspondingly. Accordingly, orange juice with a high suspended solids content due to a high content of pulp, can be significantly concentrated in the inventive apparatus to almost five times more concentrated without fouling the membrane surface. Conventional DOC or RO processes would have experience significant membrane fouling with a high suspended solids content orange juice that could not have economically produced high quality orange juice concentrate.

15 EXAMPLE 3

This example illustrates concentration of a tomato juice using the inventive pilot scale apparatus described in Example 1. Approximately 2.3 gallons of tomato juice, Brix 6.0, was obtained from Nestle. Approximately 4.0 gallons of OA (high fructose corn syrup), Brix 75.9, were used. Figure 5 shows a graph of a flux constant versus time of concentration measuring either the juice or the OA. This graph shows that the flux of water into the OA is relatively linear over time, indicating that there is very little, if any, membrane fouling.

EXAMPLE 4

This example illustrates concentration of coffee as a step in the production of instant coffee or liquid coffee concentrates. Successive coffee batches have been concentrated from 5 Brix to between 56 and 63 Brix using 60-70 Brix HFCS (high fructose corn syrup - 55% fructose, 42% glucose, 3% sucrose) as the OA. A single cell and single set of membranes was used for these experiments. The membranes were cleaned every 8 to 10 hours of use by flushing with water for 5 to 15 minutes. After this flushing, no coffee deposits were visible inside the cell (made from a clear plastic material). The cell was cleaned between batches with a 5 minute recirculation time with ULTRASIL cleaner followed by a water rinse.

The total run times for these experiments were 150 hours. Flux rates were found to vary with coffee concentration as shown in Figure 6. Flux declines with time (a measure of fouling rate) was minimal. By periodic addition of single strength coffee, 20 Brix coffee was run for 8 hours during which time no flux decline was measured. A similar run with 45 Brix coffee showed a decline of around 20% over six hours. The rates shown in Table 1 below are composite values from several batches.

It was further determined whether or not the coffee was adulterated in any way during the concentration procedure by conducting high performance liquid chromatography (HPLC) analysis to examine for sugars. No sugars passed through the membrane as shown in Table 1.

TABLE 1

	CONCENTRATION (g/100 ml)				
	unk 1	glucose	unk 2	fructose	unk 3
reference	0	0	0.0951	0.3440	0
sample					
batch 1 (56 Brix)	0	0	0.0702	0.2136	0.1273
batch 2 (40 Brix)	0	0	0	0.1515	0.1345

EXAMPLE 5

This example illustrates concentration of a raspberry juice using a HFCS OA. C13 levels in the sugars in the raspberry juice and the OA are shown in Table 2. These data show that no detectable amount of sugar passed from the OA into the juice. Table 2 below provides stable carbon isotope data of red raspberry juice and concentrates and the OA before and after processing.

TABLE 2

Stable carbon isotope data of red raspberry juice and concentrates and the OA before and after DOC processing.

SAMPLE	del ¹³ C _{PDB}
FST single strength	-24.9
FST centritherm concentrate	-24.8
Sample A	-24.8
Sample B	-24.9
OA (prior to processing)	-11.2
OA (post-process Sample A)	-11.6
OA (post-process Sample B)	-11.2

The data in Table 2 are reported relative to the PDB standard and are accurate to 0.2 mil.

EXAMPLE 6

This example illustrates the results of wine making experiments to produce a low alcohol wine. Approximately 20 gallons of red wine must (slurry of grape juice, skins, pulp and seeds) was made from ripe (22 to 23 degrees Brix) Pinot Noir grapes. The potential alcohol content of the wine, after fermentation, was about 12.4%. Ten gallons of must was immediately pressed away from the skins and seeds. The resulting juice was diluted with water, prior to fermentation, to reduce the Brix level to 11 degrees. The potential alcohol content of the wine

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after fermentation of the diluted juice would now be about 6.2%. All of the pomace (pressed skins and seeds) was re-slurried with part of the diluted juice to the original volume of 10 gallons.

After vinification, the alcohol content of the wine made from the undiluted red wine must was 12.6%, and 6.0% from the diluted must. The 12.6% alcohol wine was, for all practical purposes, a normal, high quality dry, red table wine. The 6.0% alcohol wine had good color and good aroma, but the fruit flavor of the wine was dilute and had a thinner feel in the mouth than the higher alcohol wine.

About 5 gallons of the lower alcohol wine was subject to DOC according to the inventive method and utilizing the inventive device. Both alcohol and water were removed in a constant ratio to keep the alcohol level at around 6.0%. The device used an osmotic agent (OA) of high fructose corn syrup (HFCS) diluted to 15 Brix. The resulting, concentrated, low alcohol wine was still not quite as good as the high alcohol wine, but was a profound improvement over the unconcentrated low alcohol beverage.

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EXAMPLE 7

This example illustrates the results of wine making experiments to improve the quality of a low grade wine. Approximately 5 gallons of Chardonnay wine, vinified from grapes harvested at 19 degrees Brix (harvest in a normal year would occur at 24 degrees Brix) and chaptalized to 22 degrees Brix with cane sugar prior to fermentation, was subjected to DOC, according to the inventive process and utilizing the inventive device, to remove water only. The OA was HFCS of between 50 and 70 Brix. The original wine (not subject to DOC) had an alcohol content of 12.1%, was thin and nondescript, and totally lacked any Chardonnay character. After removing 10% of the water by DOC, the alcohol content went to 13.2% and a substantial improvement was made in both flavor and textural qualities, and the wine became recognizable as a Chardonnay.

It should be noted that although this example used a low grade Chardonnay wine to illustrate the improvements than can be made, it could also have been done to concentrate the grape juice, instead of the finished wine, and then vinifying the concentrated juice.

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EXAMPLE 8

This example illustrates the results of wine making experiments to concentrate grape juice to make a desert wine without waiting for late harvest grapes. Approximately 165 gallons of Sauvignon Blanc grape juice, freshly pressed out at 27 degrees Brix, was subject to DOC (according to the inventive procedure and using the inventive device having an OA of HFCS between 50 and 70 Brix) to remove only water until the Brix level reached 40 degrees Brix. The resulting concentrated juice was diluted slightly to 37 degrees Brix with untreated Sauvignon Blanc juice. The unconcentrated juice had a potential alcohol content, after vinification, of 14.9% and little or no potential as a sweet, high alcohol, desert wine. The

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concentrated juice had a potential alcohol content, after vinification, of 20.4%. However, alcohol levels above 15% are toxic to wine yeast, so the concentrated juice had the potential for vinification into a desert wine of 15 to 16% alcohol with a residual sugar level of 8% to 10%. After vinification, the actual alcohol level was 17.4% and the residual sugar was 5.4%. The

5 wine was diluted to 15.5% alcohol with Sauvignon Blanc grape juice, which also raised the sugar level to 10.4%. The result was an extremely high quality desert Sauvignon Blanc wine.

It should be noted that the cost and juice recovery of the late harvest grape juice is many times higher than grape juice obtained from a normal harvest time. Therefore, one advantage of the present invention is an ability to use normal harvest grape juice to make a high quality desert

10 wine.

What is claimed is:

1. An osmotic concentration apparatus comprising an enclosed apparatus having outer walls, a flow inlet and outlet connected to a first chamber, an osmotic agent (OA) inlet and outlet connected to a second chamber, and a plurality of concentration cells, wherein a concentration cell comprises a first chamber defined by a semipermeable membrane, wherein the semipermeable membrane has a first side and a second side, and a second chamber wherein the second chamber is defined by the second side of the semipermeable membrane and comprises a plurality of support members arranged in parallel, whereby when fluid pressure is applied to juice within the first chamber at a pressure higher than fluid pressure in the second chamber, the semipermeable membrane deflects into the second chamber between the support members, and defining a corrugated flow path within the first chamber.
2. The osmotic concentration apparatus of claim 1 wherein the radius of curvature of the flow path in the first chamber when greater pressure is applied is from about 1.5 cm to about 4.5 cm.
3. The osmotic concentration apparatus of claim 2 wherein the radius of curvature of the flow path is about 2.0 cm.
4. The osmotic concentration apparatus of claim 1 wherein the semipermeable membrane comprises a polymeric material selected from the group consisting of hydrophobic and hydrophilic semipermeable membranes and having a thickness of from about 0.02 mm to about 0.2 mm.
5. The osmotic concentration apparatus of claim 1 wherein the distance between support members is from about 1 cm to about 10 cm.
6. The osmotic concentration apparatus of claim 1 wherein the membrane is further supported by a flexible fabric on the second side of the membrane.
7. A method for direct osmotic concentration (DOC) of a juice, comprising:
 - (a) providing a juice in a continuous flow stream to a juice inlet of an osmotic concentration cell described in claim 1;
 - (b) pumping the juice at a rate of from about 0.01 m/sec to about 2 m/sec through the first chamber in the osmotic concentration cell; and
 - (c) providing an OA in the second chamber and pumping the OA at a rate of from about 0.01 m/sec to about 1 m/sec.

8. A method for removing water from grape juice used for wine making, comprising: (a) providing a grape juice to a juice inlet in an osmotic concentration apparatus described herein; (b) providing an OA to the OA chamber, wherein the OA comprises from about 30% to about 85% by weight sugar; (c) applying greater pressure to the wine than to the OA to deflect the semipermeable membrane into a corrugated flow path; and (d) removing water from the grape juice.

9. A method for producing a low alcohol wine, comprising: (a) providing wine to a juice inlet in an osmotic concentration apparatus described herein; (b) providing an OA to the OA chamber, wherein the OA comprises from about 10% to about 30% by weight sugar; (c) applying greater pressure to the wine than to the OA to deflect the semipermeable membrane into a corrugated flow path; and (d) removing mostly alcohol from the wine.

10. A method for strengthening wine, comprising: (a) providing wine to a juice inlet in an osmotic concentration apparatus described herein; (b) providing an OA to the OA chamber, wherein the OA comprises from about 25% to about 50% by weight sugar; (c) applying greater pressure to the wine than to the OA to deflect the semipermeable membrane into a corrugated flow path; and (d) removing approximately equal percentages of alcohol and water from the wine.

11. The method of claims 9 or 10, wherein the application of pressure in step (c) is accomplished with nitrogen pressurization to avoid any unnecessary shear pressure on the wine.

12. The method of claims 9 or 10, further comprising recycling the OA by selectively distilling off alcohol and water to restore the original weight percentage of sugar in the OA.

13. The method of claims 9 or 10 wherein the OA further comprises at least 1 mM of an organic acid.

14. The method of claim 13, wherein the organic acids in the OA are selected from the group consisting of malic acid, citric acid, tartaric acid, ascorbic acid, and combinations thereof.

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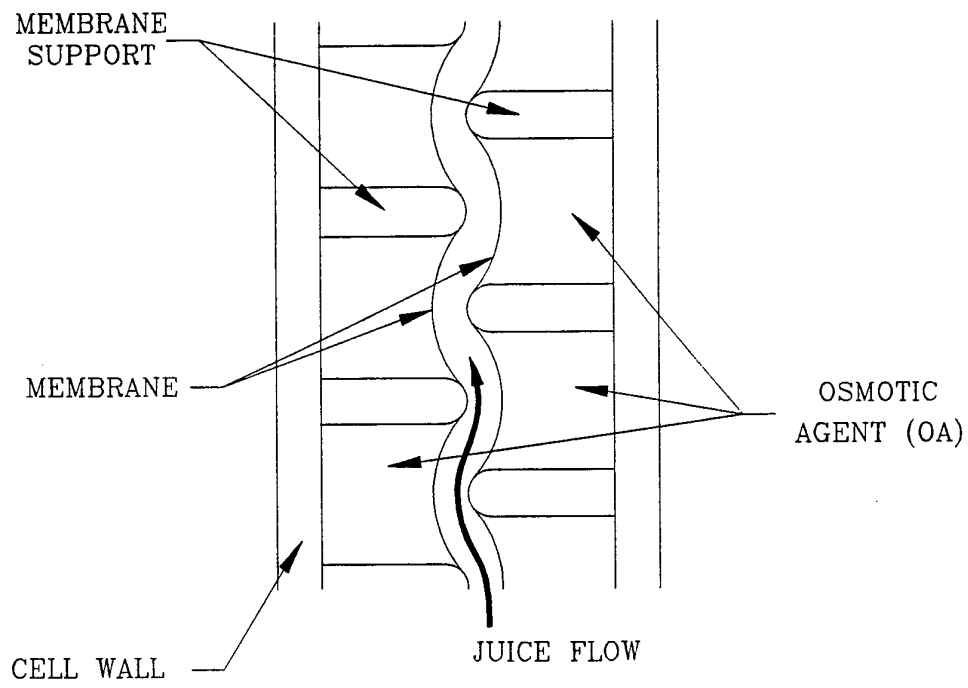


Fig. 1

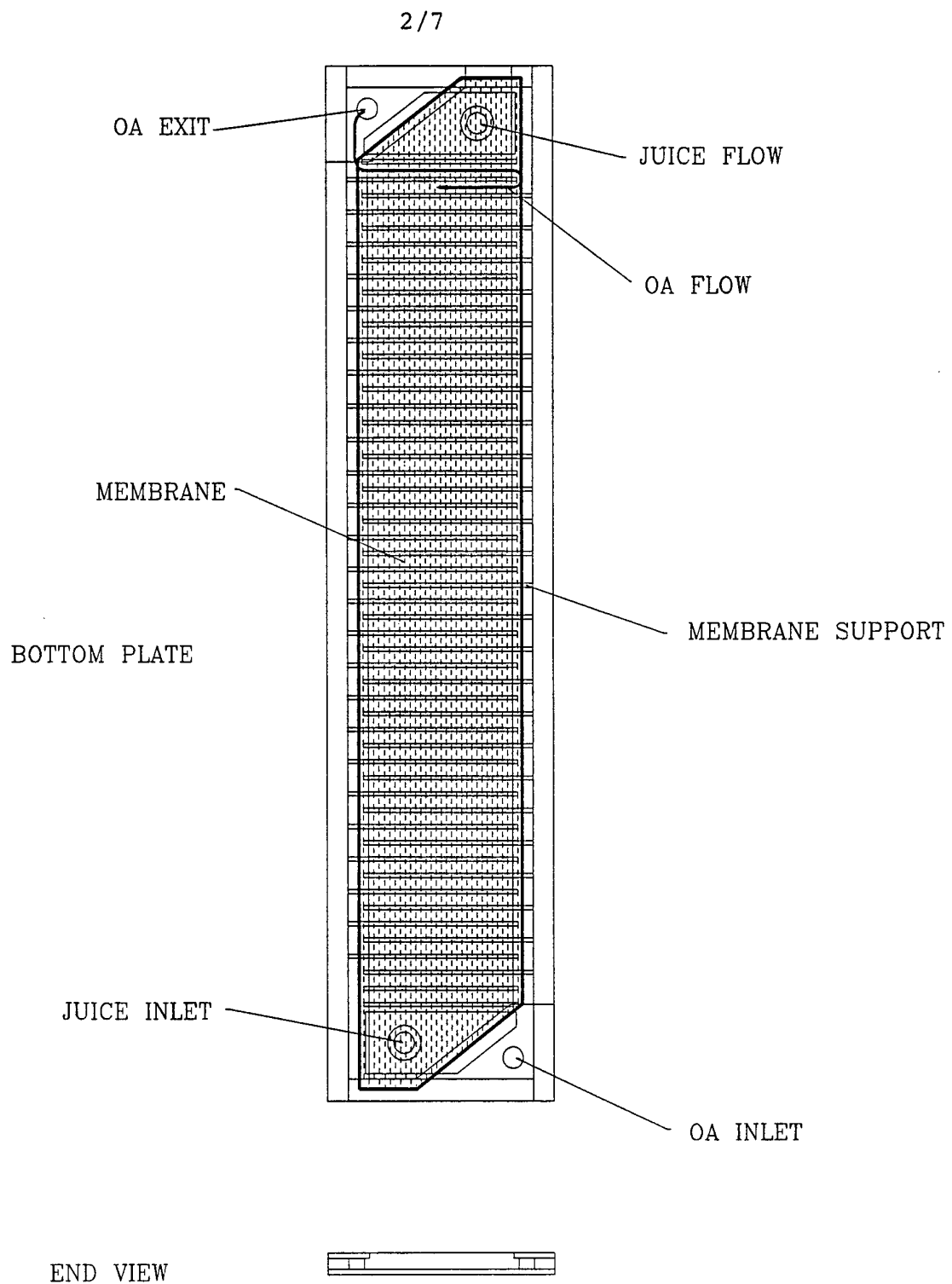


Fig. 2

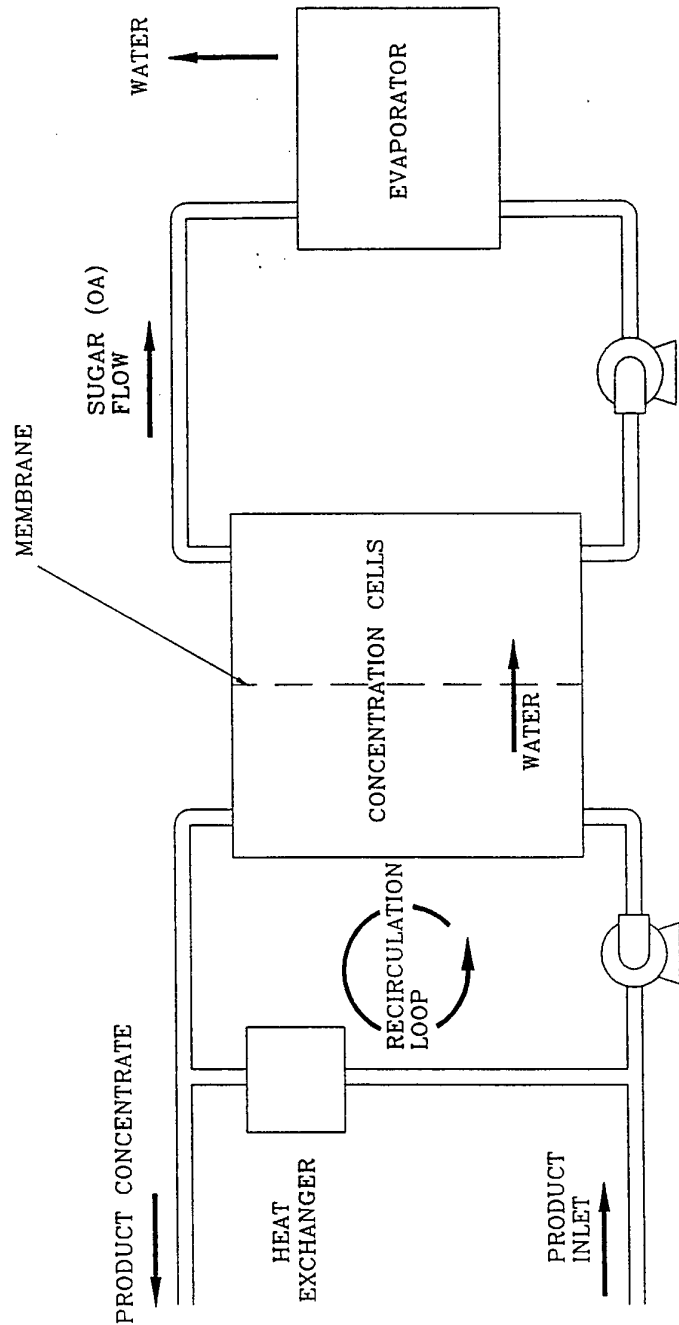


Fig. 3

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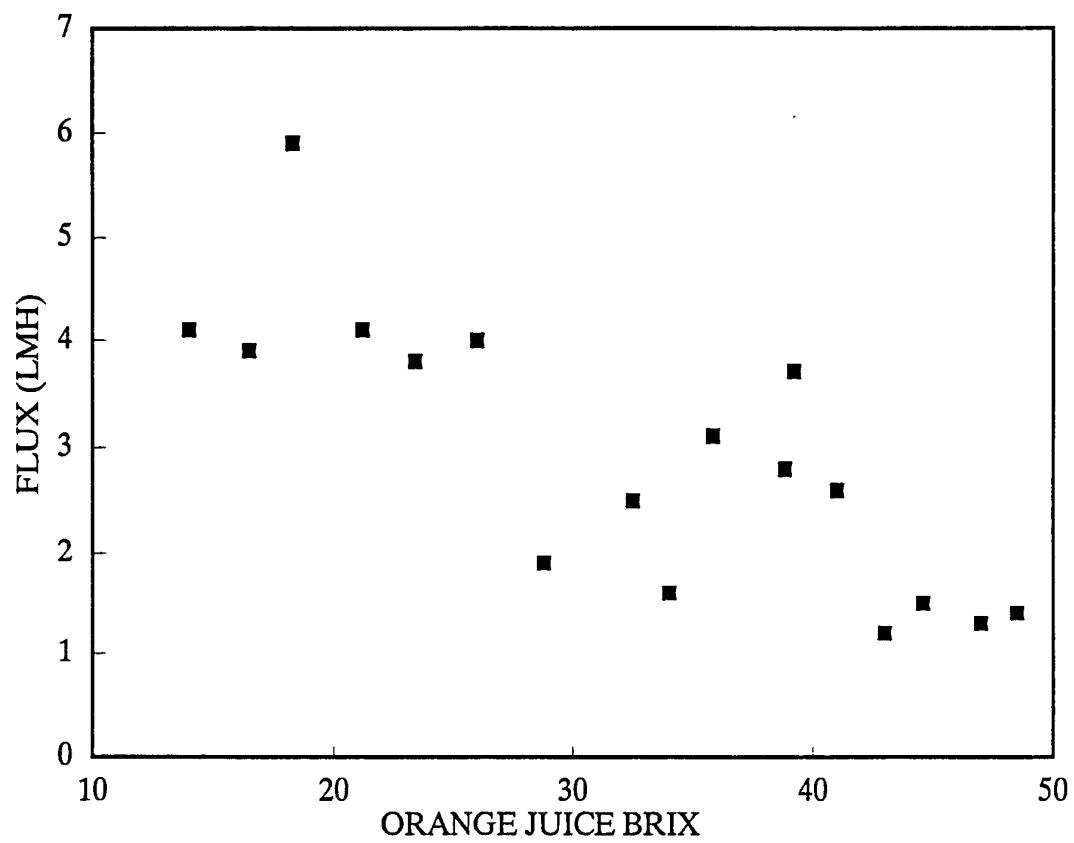


Fig. 4

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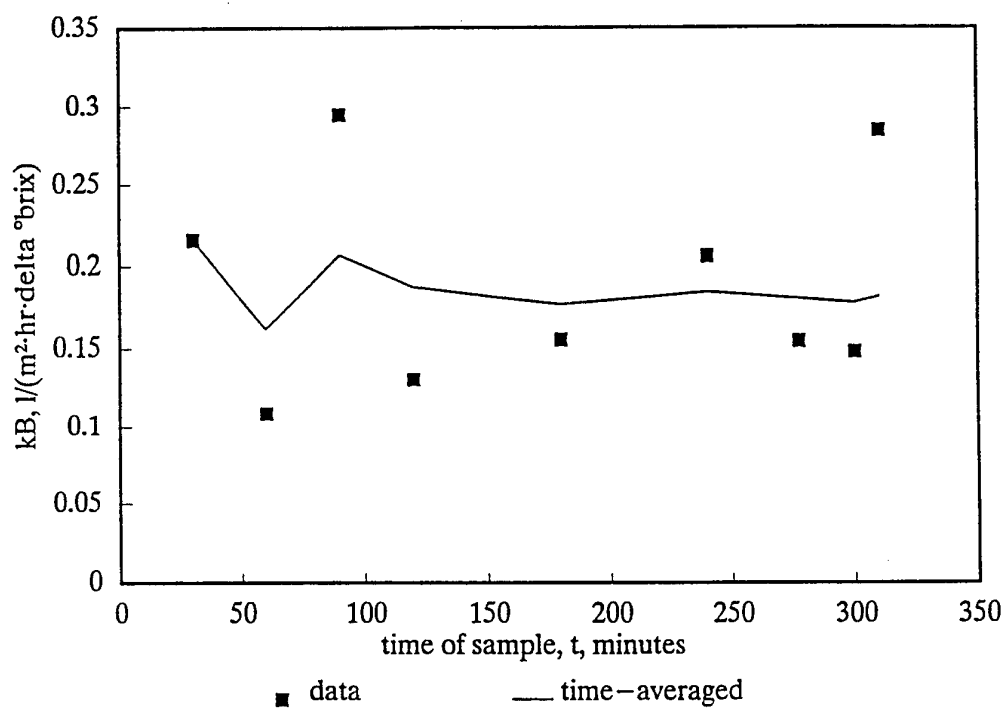


Fig. 5

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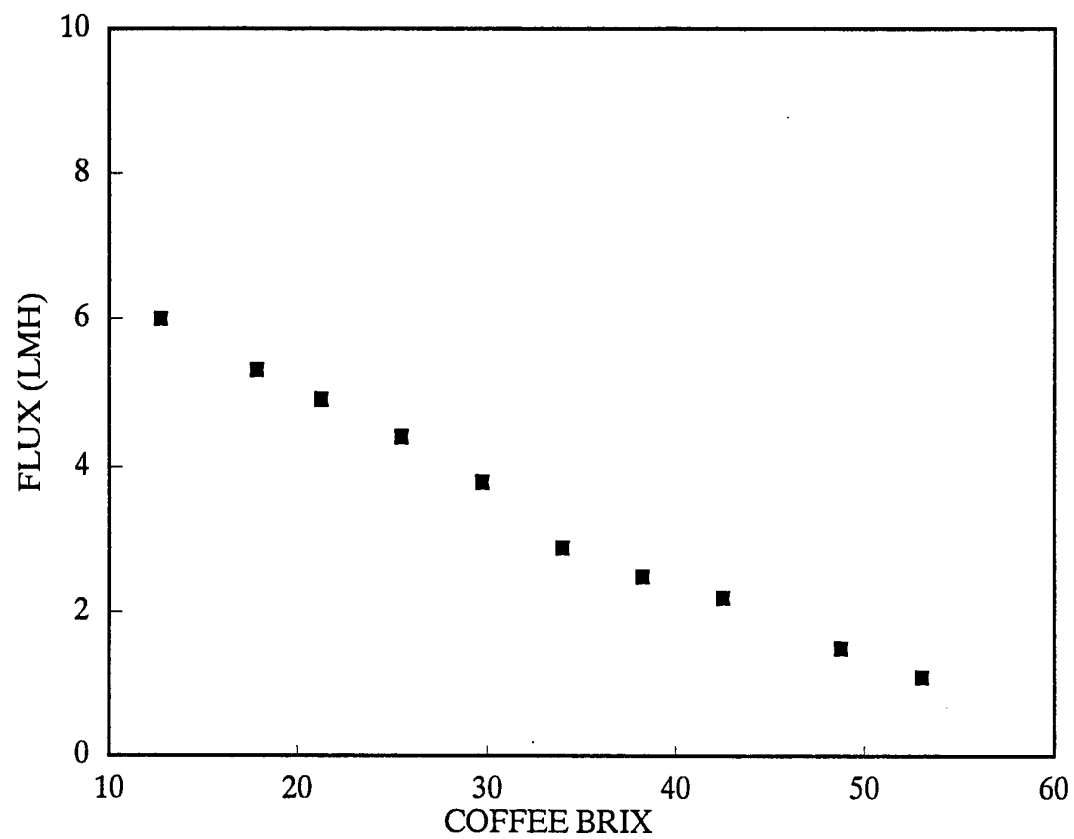


Fig. 6

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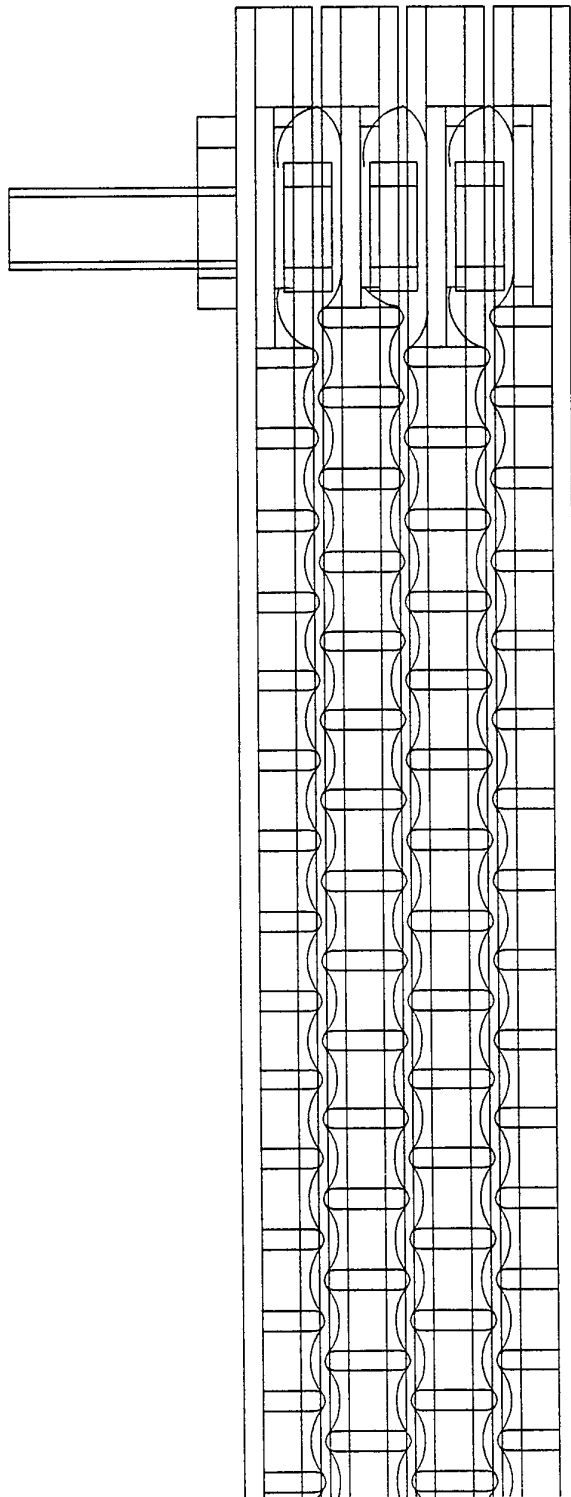


Fig. 7

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US93/11824

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) :A23P 1/00; B01D 63/00; C12G 1/00

US CL :426/490, 493, 495; 210/321.75, 644

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)

U.S. : 426/478, 592; 210/646

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 4,988,525 (GRESCH) 29 January 1991.	1-14
Y	US, A, 4,581,236 (BANDEL ET AL) 08 April 1986, see the entire document.	9, 11-14
Y	US, A, 4,664,918 (TILGNER ET AL) 12 May 1987, see the entire document.	9, 11-14
A	US, A, 4,942,045 (THUMM) 17 July 1990.	8
A	US, A, 3,052,546 (RIDDELL ET AL) 04 September 1962.	10



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:	"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
"A" document defining the general state of the art which is not considered to be part of particular relevance	"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
"E" earlier document published on or after the international filing date	"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
"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)	"&" document member of the same patent family
"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	

Date of the actual completion of the international search

31 MARCH 1994

Date of mailing of the international search report

APR 14 1994

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/US93/11824

BOX II. OBSERVATIONS WHERE UNITY OF INVENTION WAS LACKING

This ISA found multiple inventions as follows:

Group I. Claims 1-6 drawn to an osmotic concentration apparatus comprising an enclosed apparatus having outer walls, a flow inlet and outlet connected to a first chamber, an osmotic agent inlet and outlet connected to a second chamber, and a plurality of concentration cells, wherein a concentration cell comprises a first chamber defined by a semipermeable membrane, wherein the semipermeable membrane has a first side and a second side, and a second chamber wherein the second chamber is defined by the second side of the semipermeable membrane and comprises a plurality of support members arranged in parallel, classified in Class 210, subclass 321.75.

Group II. Claim 7, drawn to a method for direct osmotic concentration of a juice using the osmotic concentration cell described in claim 1, classified in Class 426, subclass 478.

Group III. Claim 8, drawn to a method for removing water from grape juice using an osmotic concentration apparatus and an osmotic agent comprises about 30% by weight sugar, classified in Class 426, subclass 495.

Group IV. Claims 9-14 drawn to a method for treating wine with an osmotic concentration apparatus and an osmotic agent comprises about 30% by weight sugar to produce a low alcohol wine or to strengthen the wine, classified in Class 426, subclass 493.

Unity exists between the claims of Groups I and II where the common special technical feature is the osmotic concentration cell described in claim 1, or between the claims of Groups III and IV where the common special technical feature is the osmotic agent comprises about 30% by weight sugar. However, unity is lacking between the claims of Groups I and II the claims of Groups III and IV since the claims of Groups I and II do not include the same or corresponding special technical feature as set forth in the claims of Groups III and IV, i.e., an osmotic agent comprises about 30% by weight sugar.

Note that because Group I would be examined with Group II and because Group III would be examined with Group IV, there are only two groups of claims on which this requirement is based. Accordingly, a payment of \$170.00 provides for the search report to cover all the claims.