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(54) Sugar beet membrane filtration process

(57) The present invention relates to a process for producing sugar from beets, comprising the steps of:
(f) macerating sugar beets or pieces thereof, thereby producing a macerated material that comprises pulp and liquid,
(g) separating the liquid in the macerated material from the pulp at a temperature of at least about 65° C to produce a sucrose-containing feed juice;
(h) filtering the sucrose-containing feed juice through a first ultrafiltration membrane that has a first molecular weight cutoff, thereby producing a first ultrafiltration permeate and a first ultrafiltration retentate;
(i) filtering the first ultrafiltration permeate through a second ultrafiltration membrane that has a second molecular weight cutoff that is lower than the first molecular weight cutoff, thereby producing a second ultrafiltration permeate and a second ultrafiltration retentate; and
filtering the second ultrafiltration permeate through a nanofiltration membrane, thereby producing a nanofiltration permeate and a nanofiltration retentate, wherein the nanofiltration retentate has a higher concentration of sucrose on a dry solids basis than the feed juice in step (c).

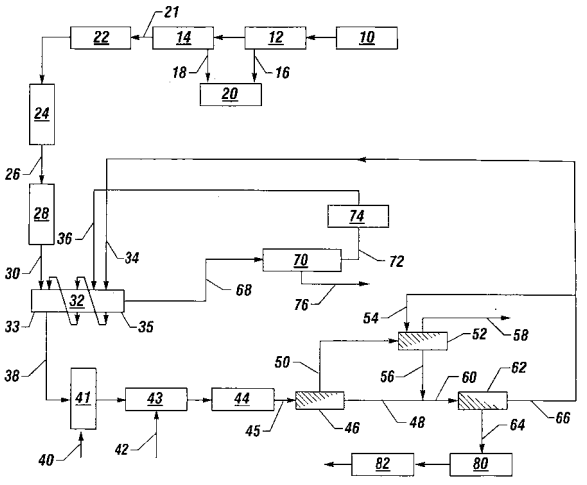

FIG. 1

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Description

BACKGROUND OF THE INVENTION

[0001] The present application is a divisional application of EP00957444.3. The present invention relates to a process for producing sucrose from sugar beets.

[0002] The conventional beet sugar manufacturing process involves cleaning the beets, slicing them into cossettes, extracting juice from the cossettes by diffusion, purifying the juice by liming and carbonation, concentrating the juice by multiple effect evaporation, multi-stage boiling of concentrated juice in pans, separation, washing, and drying the sugar.

[0003] Juice extraction in the conventional process is done by allowing the sugar to diffuse through the natural cell walls of beets. The cell walls allow sugars and other low molecular weight compounds to pass through but prevent the passage of high molecular weight compounds. This selective diffusion process has two advantages. Retaining the high molecular weight compounds helps produce a high purity juice. It also reduces filtration difficulties that are caused by polysaccharides and proteins that comprise the high molecular weight compounds.

[0004] Purification of beet juice in the conventional process is based on lime treatment. Lime serves many purposes in the juice purification process. It neutralizes the acidity of the juice and precipitates calcium salts of several organic and inorganic acids. The precipitate absorbs other impurities. The lime precipitate produces a porous mass, which facilitates subsequent filtration of juice.

[0005] The conventional diffusion process for juice extraction from beets has disadvantages. It has a long retention time, which encourages microbial growth, resulting in sugar loss and formation of undesirable compounds. It is difficult to increase the temperature of sliced cossettes quickly enough to prevent growth of micro-organisms. Typically the pulp remaining after diffusion is pressed and the press juice is introduced back into the diffuser. A significant portion of the high molecular weight compounds retained by the cell walls in the diffusion process is released in pressing to be mixed with the diffusion juice. This partially negates the advantages of the selective diffusion process.

[0006] The conventional liming process uses large quantities of lime, amounting to about 2.5% of the total weight of beets processed. Beet sugar plants operate lime kilns and transport limestone over long distances for this purpose. The effluent from the liming-carbonation process, consisting of used lime and separated impurities, is disposed as waste. Production of lime and disposal of liming effluent are costly operations. Disposal of liming effluent is becoming increasingly difficult and expensive in many communities.

[0007] Conventional dead-end filtration is incapable of separating sucrose from macromolecular impurities in beet juice. Several methods of using microfiltration and ultrafiltration for purification of juice with reduced lime use have been reported, but these methods generally involve inserting microfiltration or ultrafiltration membranes into the conventional beet process at one or more points.

[0008] There is a long-standing need for improved processes for obtaining sugar from beets that avoid or at least minimize one or more of the problems existing in the previously used processes.

SUMMARY OF THE INVENTION

[0009] The present invention relates to a process for producing sugar from sugar beets. In one embodiment, the process comprises the steps of (a) either (i) macerating beets or pieces thereof or (ii) slicing beets into cossettes; (b) separating juice from the macerated beets or the cossettes; and (c) membrane filtering the separated juice, producing a retentate and a permeate.

[0010] In another embodiment, the process comprises the steps of: (a) macerating beets or pieces thereof; (b) mechanically separating juice from the macerated beets; and (c) membrane filtering the separated juice, producing a retentate and a permeate. This embodiment of the present invention makes use of mechanical means, such as vacuum filtration, for separating juice from macerated beets, as opposed to the simple diffusion process that is used in prior beet processing technology to obtain juice from cossettes.

[0011] In certain preferred embodiments of the process, where beets are cut into pieces and subsequently macerated, and the maceration is done in an attrition mill. It is also preferred that vacuum extraction of juice is done on a moving porous filtration belt with countercurrent flow of macerated beets and water, most preferably at a temperature of at least about 80 °C. The pH of the vacuum extracted juice preferably is adjusted to at least about 7 by addition of sodium hydroxide.

[0012] In one preferred embodiment of the process, the extracted juice is contacted with an agent selected from the group consisting of sulfur dioxide, sulfate salts, sulfite salts, bisulfite salts, and mixtures thereof, in an amount sufficient to adjust the pH of the extracted juice to no greater than about 8.

[0013] The membrane filtration can suitably be done with an ultrafiltration membrane, a nanofiltration membrane, or other types of membranes described herein. In one preferred embodiment, the membrane filtration is cross-flow ultrafiltration, and is done at least about 80°C, and the pH of the permeate is at least about 7.

[0014] One preferred option in the process is to subject the retentate from the membrane filtration to diafiltration, in

order to recover residual sugar in the retentate, thereby producing a diafiltration filtrate (also referred to herein as diafiltrate). This diafiltrate preferably is combined with the membrane filtration permeate for further processing.

[0015] Another preferred option in the process is concentration of the permeate from the membrane filtration by reverse osmosis, thereby producing a concentrated solution. This concentrated solution is evaporated and sucrose is crystallized therefrom.

[0016] Preferably in the process of the present invention no lime and no carbon dioxide are contacted with the juice or the permeate.

[0017] One specific preferred embodiment of the process comprises the steps of: (a) cutting sugar beets into pieces; (b) macerating the beet pieces; (c) mechanically extracting juice from the macerated beets; (d) sulfitation of the extracted juice; (e) pH adjustment of the extracted juice to at least about 7; (f) membrane filtering the extracted juice, producing a retentate and a permeate; (g) subjecting the retentate to diafiltration, thereby producing a diafiltration filtrate that is enriched in sugar compared to the retentate; (h) combining the diafiltration filtrate and the permeate from the membrane filtration, thereby producing a combined juice; (i) concentrating the combined juice by reverse osmosis, thereby producing a concentrated solution; and (j) evaporating the concentrated solution and crystallizing sucrose therefrom.

[0018] This embodiment of the present invention has many advantages over the conventional process using diffusion, liming and carbonation. For instance, this process has a lower retention time, which reduces the extent of microbial destruction of sucrose. The fineness of the macerated beets reduces the percentage of sucrose retained in the pulp to below about 0.5% compared to as high as 0.75% in the conventional process. Higher extraction due to maceration and reduction in inversion due to reduced retention time increase the total sugar recovery by about 1 to 2% of the weight of beets processed.

[0019] This method of purification produces a beet juice of lower color than the traditional diffusion and carbonation process. Less color in the juice allows for less washing of the final crystalline product. Membrane filtration removes macromolecules in the beet juice, producing syrups of lower viscosity. Lower viscosity syrups crystallize faster and purge easier from the sucrose crystal surface. Low color, low viscosity syrup, reduces recycle during the crystallization process, resulting in better sugar recovery.

[0020] The process eliminates the lime kiln, lime quarries and all associated equipment, processes, products, by-products and waste products. Sodium hydroxide for neutralization of juice costs about 50% less than the lime that it replaces. Sodium hydroxide is easier to handle, cleaner and less abrasive on equipment than lime.

[0021] Also, the present invention results in a drastic reduction of waste products that cause environmental pollution. The conventional process produces a filter cake that comprises products of the liming process and impurities removed from the juice. This cake is disposed into ponds or landfills. The proposed process completely eliminates the need for disposal of such materials. Invert sugars end up with the molasses which is a salable byproduct and not in the effluent. The present invention also allows elimination of the carbonation process, which is a major source of atmospheric pollution in beet sugar plants.

[0022] In another embodiment of the present invention, a sucrose-containing feed juice that has been obtained from sugar beets is filtered through a first ultrafiltration membrane that has a first molecular weight cutoff. This ultrafiltration step produces a first ultrafiltration permeate and a first ultrafiltration retentate. The first ultrafiltration permeate is filtered through a second ultrafiltration membrane that has a second molecular weight cutoff that is lower than the first molecular weight cutoff. This second ultrafiltration step produces a second ultrafiltration permeate and a second ultrafiltration retentate. The second ultrafiltration permeate is nanofiltered through a nanofiltration membrane, thereby producing a nanofiltration permeate and a nanofiltration retentate. The nanofiltration retentate has a higher concentration of sucrose on a dry solids basis than the feed juice introduced into the first ultrafiltration step, and can be used in evaporation and crystallization operations to produce crystals of white sugar.

[0023] In one particular embodiment of the invention, the sucrose-containing feed juice is manufactured by macerating sugar beets or pieces thereof, thereby producing a macerated material that comprises pulp and liquid, and then separating the liquid in the macerated material from the pulp, for example by one or more of centrifugation, conventional filtration, or screening. In one particular embodiment, the beets are macerated by first passing them through a hammer mill, and optionally they can subsequently be passed through a grinder, whereby the beets are converted into a mixture of pulp and sucrose-containing liquid. Preferably, no more than about 5 % by weight of the sucrose present in the beets remains in the pulp after the liquid is separated therefrom, more preferably no more than about 3 %.

[0024] After separation of the fibrous pulp from the liquid, and before the first ultrafiltration, the process can optionally include an additional step or steps to remove residual beet fibers and silt from the separated liquid (juice). This can be done by screening and/or filtration. Preferably the screening or filtration removes at least 90 % by weight of all fibers and silt having a largest dimension of about 150 μm or greater, more preferably at least 90 % by weight of all fibers and silt having a largest dimension of about 50 μm or greater.

[0025] In another embodiment of the invention, instead of macerating beets, they are sliced into cossettes and the sucrose-containing feed juice is obtained therefrom by diffusion.

[0026] It is also possible to introduce air into the feed juice prior to the first ultrafiltration, in order to oxidize color-

forming materials. This oxidation, while increasing the color of the juice, causes the color-forming materials to polymerise, which facilitates their removal in the subsequent ultrafiltration. (When this patent refers to polymerisation of color-forming materials, this is intended to include physical agglomeration as well as chemical polymerisation.) Another option is to introduce hydrogen peroxide, ozone, or both, into the feed juice prior to the first ultrafiltration. These materials also facilitate oxidation.

[0027] It is preferred to adjust the pH of the feed juice to about 6-8, for example by the addition of a base, prior to ultrafiltration. This can help minimize formation of invert.

[0028] The first ultrafiltration membrane preferably has a molecular weight cutoff of at least about 2,000 daltons and a pore size no greater than about 0.1 microns. More preferably, it has a molecular weight cutoff of about 4,000-200,000 daltons. The first ultrafiltration permeate preferably has a color of about 3,000-10,000 icu. (All color values given herein are determined on an ICUMSA scale.)

[0029] The process of the present invention can be operated at a number of different process conditions. As representative examples of such conditions, the feed juice can be at a temperature of about 140-200 °F (60-93 °C) during the first ultrafiltration, more preferably about 160-185 °F (71-85 °C).

[0030] The second ultrafiltration membrane preferably has a molecular weight cutoff of about 500-5,000 daltons, more preferably about 1,000-4,000 daltons. In one particular embodiment of the process, the second ultrafiltration is performed in two stages, the first stage using an ultrafiltration membrane having a molecular weight cutoff of about 3,500-4,000 daltons, and the second stage using an ultrafiltration membrane having a molecular weight cutoff of less than about 3,500 daltons. The second ultrafiltration permeate preferably has a color no greater than about 4,000 icu, more preferably no greater than about 2,500 icu.

[0031] In order to minimize loss of sucrose in the retentate from the first and second ultrafiltration steps, it is preferable to include diafiltration steps in the process. "Diafiltration" is used herein to mean ultrafiltration that employs added water in the feed to help flush sucrose through the membrane.

[0032] In one such embodiment of the process, the first ultrafiltration retentate is diafiltered through at least a first diafiltration/ultrafiltration membrane. This produces a first diafiltration permeate and a first diafiltration retentate. The first diafiltration permeate is then combined with the first ultrafiltration permeate and filtered through the second ultrafiltration membrane.

[0033] Similarly, the retentate from the second ultrafiltration can be diafiltered through at least a second diafiltration/ultrafiltration membrane. This second diafiltration step produces a second diafiltration permeate and a second diafiltration retentate. The second diafiltration permeate is then combined with the second ultrafiltration permeate and subsequently filtered through the nanofiltration membrane.

[0034] The retentates from the first and second ultrafiltrations (or diafiltrations) and the nanofiltration permeate can be combined to produce molasses. This combined stream may need to be concentrated by evaporation of water.

[0035] In addition to purification of the juice by nanofiltration, it is possible to include in the process ion exchange and/or electrodialysis purification steps. These three purification methods can be used in any sequence. In one particularly preferred embodiment of the process, the nanofiltration retentate is purified by electrodialysis, thereby producing a electrodialyzed juice and an electrodialysis residue, and then the electrodialyzed juice is purified by ion exchange, thereby producing a purified juice. Preferably, no lime and no carbon dioxide are contacted with any of the permeates.

[0036] The nanofiltration removes ash (including mono- and divalent cations), invert, organic acids, nitrogenous material and other low molecular weight organic or charged compounds. The nanofiltration and the optional electrodialysis and/or ion exchange preferably remove at least about 65 % by weight of the Ca, Mg, K, Na and their associated inorganic and organic anions that are present in the second ultrafiltration permeate. The ion exchange replaces remaining divalent cations such as calcium and magnesium with monovalent cations such as potassium and sodium. Preferably, the nanofiltration retentate has a lower concentration of divalent cations on a dry solids basis than the second ultrafiltration permeate.

[0037] The nanofiltration permeate will contain a large percentage of the impurities that were present in the feed juice. For example, in many instances, the nanofiltration permeate will comprise at least about 30% by weight on a dry solids basis of the ash, at least about 30% of the invert, and at least about 25% of the betaine present in the feed juice.

[0038] The purified juice (i.e., after nanofiltration and any electrodialysis and/or ion exchange), preferably has an ash concentration of no greater than about 2.5 % by weight on a dry solids basis, more preferably no greater than about 2 %, most preferably no greater than about 1.0 %.

[0039] After the membrane filtration steps (and any electrodialysis and/or ion exchange), water can be evaporated from the purified juice to produce a concentrated syrup (e.g., 75% dry solids). White sugar can then be crystallized from the concentrated syrup. Because of the high degree of removal of impurities, the present invention can achieve two crystallizations of white sugar from the concentrated syrup, as opposed to one in typical prior art beet processes.

[0040] A mother liquor will remain after one or more crystallizations of white sugar from the concentrated syrup. This mother liquor can be recycled to one of the ultrafiltrations. Optionally, this recycle stream can be further purified to reduce its raffinose content.

[0041] The process can optionally include sulfitation of one or more process streams. In particular, at least one aqueous stream selected from the group consisting of the feed juice, the first ultrafiltration permeate, the second ultrafiltration permeate, the nanofiltration retentate, and the evaporator feed can be contacted with an agent selected from the group consisting of sulfur dioxide, sulfite salts, bisulfite salts, metabisulfite salts, dithionites, and mixtures thereof, in an amount sufficient to provide an equivalent concentration of sulfur dioxide in the stream of at least about 100 ppm.

[0042] One particularly preferred embodiment of the invention is a process for producing sugar from beets that comprises the steps of:

(a) macerating sugar beets or pieces thereof, thereby forming pulp that comprises sucrose-containing aqueous liquid;
(b) separating the sucrose-containing liquid from the pulp (or, as an alternative to steps (a) and (b), slicing sugar beets into cossettes and obtaining a sucrose-containing feed juice therefrom by diffusion);
(c) filtering the sucrose-containing liquid through a first ultrafiltration membrane that has a molecular weight cutoff of about 4,000-200,000 daltons, thereby producing a first ultrafiltration permeate that has a color no greater than about 10,000 icu and a first ultrafiltration retentate;
(d) filtering the first ultrafiltration permeate through a second ultrafiltration membrane that has a molecular weight cutoff of about 2,000-4,000 daltons, thereby producing a second ultrafiltration permeate that has a color no greater than about 4,000 icu and a second ultrafiltration retentate;
(e) filtering the second ultrafiltration permeate through a nanofiltration membrane; thereby producing a nanofiltration permeate and a nanofiltration retentate, wherein the nanofiltration retentate has a higher concentration of sucrose on a dry solids basis than the sucrose-containing liquid in step (b);
(f) purifying the nanofiltration rententate by at least one method selected from the group consisting of ion exchange and electrodialysis, thereby producing an evaporator feed;
(g) evaporating water from the evaporator feed to produce a concentrated syrup; and
(h) crystallizing white sugar from the concentrated syrup.

[0043] Optionally, this embodiment of the process can further comprise the steps of:

(i) crystallising a mother liquor from the first crystallisation to produce white sugar;
(j) treating the mother liquor from the second crystallisation by chromatographic separation or by an enzyme to remove raffinose; and
(k) recycling the treated mother liquor back to the nanofiltration feed or the evaporator feed.

[0044] Another aspect of the present invention is a process for purifying a sucrose-containing juice obtained from sugar beets. This process comprises the steps of: (a) introducing sufficient air into the juice to cause polymerisation of color bodies; and (b) removing at least some of the color bodies from the juice by membrane filtration through at least one ultrafiltration membrane or nanofiltration membrane.

[0045] The various aspects of the present invention have a number of advantages over prior art beet processes. For example, the process of the present invention eliminates the need for a lime kiln, lime quarries and all associated equipment, processes, products, by-products and waste products. Also, the present invention results in a drastic reduction of waste products that cause environmental pollution. The conventional process produces a filter cake that comprises products of the liming process and impurities removed from the juice. This cake is disposed into ponds or landfills. The proposed process completely eliminates the need for disposal of such materials. The present invention allows elimination of the carbonation process, which is a major source of atmospheric pollution in beet sugar plants.

[0046] The present invention provides a cost-effective way of reducing the ash content of the beet juice or syrup, preferably to about 2 % or less (on a dry solids basis), more preferably to about 1.5 % or less, most preferably to about 1 % or less. This reduction in ash content is important because it allows a second strike of sucrose crystals from the syrup. In prior art beet processes, ash contents in the range of 3.5 % made it practically impossible to have more than one strike of sucrose crystals.

[0047] In addition, the present invention can eliminate the need for desugarization of molasses streams. The efficient membrane filtration steps prevent excessive amounts of sucrose from entering the molasses streams in the first place.

[0048] Further, the present invention provides an economical and reliable method for removing color-causing materials from beet juice. It also can reduce the formation of undesirable crystalline forms due to the presence of excessive amounts of raffinose.

BRIEF DESCRIPTION OF THE DRAWINGS

[0049]

Figure 1 is a process flow diagram showing a process of the present invention for obtaining sucrose from sugar beets, wherein the process comprises maceration of beets.

Figure 2 is a process flow diagram with a mass balance for another embodiment of the invention.

Figures 3 and 4 are process flow diagrams showing embodiments of the present invention in which sugar beets are macerated.

Figures 5 and 6 are process flow diagrams showing embodiments of the present invention in which sugar beets are sliced into cossettes.

DETAILED DESCRIPTION OF SPECIFIC EMBODIMENTS

[0050] The present invention provides an improved method for obtaining sucrose from sugar beets. One embodiment of the invention is represented in Figure 1.

[0051] Beets received from the field are kept in a storage area 10. Fresh beets are typically used in the process, but frozen beets can also be used. Beets from the storage 10 area are flumed to a conventional beet washing apparatus 12, in which dirt is removed from the exterior of the beets. Washed beets exiting the washing apparatus pass through a conveyor 14, where water is removed. Wash water 18 and flume water 16 streams collected from this apparatus are sent to waste water treatment ponds 20.

[0052] The washed beets 21 are carried by conveying apparatus 22 to cutting apparatus 24, such as a hammer mill or slicer, in which the beets are cut into pieces, for examples pieces having an average size of about one inch thickness. The stream of beet pieces 26 from the slicer (or alternatively the whole beets 21) are fed to macerating apparatus 28. The macerating apparatus can comprise, for example, one or more hammer mills (fixed blade type being the preferred option) that uses a set of rotating blades mounted on a horizontal shaft which forces the beet material through a discharge screen. Another macerating apparatus can comprise one or more attrition mills that use discs as the primary attrition device. The discs preferably have grooves therein to facilitate maceration, and the discs can be horizontal or vertical in positioning. Disc-type attrition mills are presently preferred over hammer mills, although it is possible to use both in series (e.g., hammer mill followed by disc attrition mill). Preferably extracted juice 38 or water 34 is fed to the macerator 28 to facilitate discharge of macerated beets and/or to control the temperature of the equipment.

[0053] The stream of macerated beets 30 is fed to a vacuum juice extraction apparatus 32. This apparatus can comprise a horizontal, porous, moving belt that is subjected to a vacuum from the bottom. Macerated beets are introduced as a uniform layer at one end (the feed end) 33 of the belt. A clean water stream 34 is introduced at the opposite or discharge end 35 of the belt. Thus, the macerated beet feed and the water feed to this apparatus 32 are countercurrent to each other. A stream of juice 36 is reintroduced over the belt, preferably at several locations. This method of countercurrent filtration produces a pulp stream 68 with low sugar content and an extracted juice stream 38 with high sugar content. The countercurrent vacuum filtration process preferably is carried out at an elevated temperature of about 80°C to control microbial growth and to improve the extraction of juice.

[0054] A centrifugal separator or a series of centrifugal separators may also be used to separate the juice 38 from the macerated beet material 68. The centrifugal separator may consist of either a vertical or horizontal rotating perforated basket in which the macerated beet material 30 is introduced into the basket and the solid phase 68 and liquid phase 38 is separated across a screen using centrifugal force. Wash water 66 and/or countercurrent extracted juice 36 is sprayed onto the macerated beet material during centrifugation to minimize sugar content in the pulp 68.

[0055] The pulp 68 leaving the juice extractor 32 has a very low sucrose content but a high water content. It is pressed in a screw press 70 to extract a dilute press juice 72 which contains about 1% dissolved solids and about 99% water. The dissolved solids comprise about 50% sucrose and 50% non-sugars. This dilute press juice 72 is raised to a temperature of about 80°C in a heater 74 and then is returned to the juice extractor 32 as stream 36. Pressed pulp 76 is used as animal feed, with or without further drying.

[0056] The extracted juice 38 is sent to tank 41 and can optionally be sulfitated by the addition of sulfur dioxide, or sulfite or bisulfite salts in a stream 40, e.g. sulfur dioxide gas or aqueous ammonium bisulfite at about 65% concentration. Preferably the residual level of sulfur dioxide in the juice after sulfitation is at least 100 ppm. The sulfitation can take place at the time of slicing, macerating, juice extraction, or other points in the process, as an alternative to or in addition to the particular sulfitation step in this embodiment. This sulfitation will prevent the color increase that can otherwise take place during subsequent membrane filtration and evaporation operations. Other antioxidants may also be used.

[0057] The juice is then neutralized by the addition of aqueous sodium hydroxide 42, preferably to a pH of at least 7, in neutralization tank 43. This pH adjustment helps prevent the inversion of sugars which takes place at elevated temperatures. Other chemicals may be also be used for pH adjustment, e.g. liquid potassium hydroxide or granular sodium carbonate.

[0058] The juice extracted from the macerated beets by the countercurrent filtration process comprises about 0.2% suspended solids, about 14% dissolved solids, and about 84% water. The dissolved solids comprise about 85% sucrose and 15% non-sugars. Preferably the temperature of the extract is about 80°C and its pH is at least 7.

[0059] The treated juice can then be passed through a heater 44 to increase its temperature to about 80°C.

[0060] The heated juice is then processed by membrane filtration 46, preferably by cross-flow ultrafiltration, to separate high molecular weight compounds from sucrose solution. Ultrafiltration produces an ultrafiltrate (also referred to as permeate or clarified juice) 48 which is about 12% dissolved solids and about 88% water. The dissolved solids comprise about 90% sucrose and 10% non-sugars. The ultrafiltrate 48 preferably has a temperature of about 80°C and its pH is at least 7.

[0061] The permeate from ultrafiltration has a sucrose purity equivalent to the thin juice produced by the conventional beet process, which is around 90%. However, there are important differences between the non-sugars in the two products. Ultrafiltered juice may contain a higher level of invert sugar and/or a lower level of macromolecular compounds than the conventional thin juice.

[0062] Invert sugars in the ultrafiltered juice will primarily end up in the molasses without reducing sucrose recovery drastically. This is an advantage compared to the conventional liming process, which sends reaction products of lime and invert sugars to the effluent disposal system. Lower levels of macromolecular compounds result in juice with lower viscosity, which has more favorable sugar boiling characteristics.

[0063] Ultrafiltration produces a juice with reduced color. The extracted juice 38 typically has color value over 100,000 on a ICUMSA scale. The ultrafiltrate 48 typically has a color value below 2,000 on the same scale. This is equivalent to or better than the color value of thin juice prepared by the conventional method. Lower color in combination with lower viscosity result in an easier sugar boiling process. The results are higher sugar extraction, more efficient sugar boiling, and lower sugar loss to molasses.

[0064] A variety of membrane configurations can be used in the present invention, including for example spiral, hollow fiber, and tubular membranes. Membranes suitable for this separation process should have two unique characteristics. They should have high permeability to water and sucrose but have low passage of colorants and other macromolecular compounds. Tight ultrafiltration membranes with a molecular weight cutoff between about 1,000 and 10,000 and loose nanofiltration membranes with NaCl rejection of about 10% are well suited for this application. Membranes that have a negative surface charge are preferred since most compounds to be rejected are negatively charged.

[0065] The retentate 50 from the ultrafiltration process contains mostly suspended and dissolved impurities. It also contains a significant amount of sucrose. In order to recover at least some of this sucrose, the retentate is diafiltered through a membrane system 52 with addition of water 54. This diafiltration extracts most of the sugar left in the ultrafiltration retentate. The diafiltrate 56 contains about 3% dissolved solids and about 97% water. The dissolved solids in the diafiltrate comprise about 88% sucrose and 12% non-sugars. Preferably the temperature of the diafiltrate is about 80°C and its pH is above 7. The retentate 58 of the diafiltration process contains about 5% suspended solids, 3% dissolved solids and about 87% water. This is concentrated by evaporation and used as animal feed, with or without mixing with pressed pulp.

[0066] The ultrafiltrate 48 and diafiltrate 56 are combined to form a composite product stream 60. The composite product stream (also referred to as purified juice) contains about 11% dissolved solids and about 89% water. The dissolved solids comprise about 90% sucrose and 10% non-sugars.

[0067] A reverse osmosis membrane system 62 may be used for pre-concentration of the purified juice stream. This is another cross-flow membrane process that is less energy intensive and more economical for pre-concentration of dilute sucrose solutions than the conventional process steps. The product 64 of the reverse osmosis system contains about 20% dissolved solids and about 80% water. The dissolved solids comprise about 90% sucrose and 10% non-sugars.

[0068] The permeate 66 of the reverse osmosis is high quality water. A portion 34 of this water is used in the countercurrent vacuum filtration process 32 and remainder in other plant applications, such as water feed 54 to the diafiltration process 52.

[0069] The temperature of the pre-concentrated sucrose solution 64 is then raised in a heater 80 and subsequently the remaining water is removed in evaporators 82. Sucrose is crystallized as in conventional processes.

[0070] Some of the equipment used in the process of Figure 1 is conventional and well known to persons of ordinary skill in this field, such as beet washing equipment, pulp presses, and evaporators. Beet slicing apparatus 24 and macerating apparatus 28 are commercially available from suppliers such as H. Putsch GmbH & Company (Hagen, Germany), Maguin Company (Charmes, France), Dakota Machine Inc. (West Fargo, North Dakota), and The Fitzpatrick Company (Elmhurst, Illinois). Suitable vacuum belt juice extraction apparatus is available from EIMCO Company (Salt Lake City, Utah), and Dorr-Oliver (Milford, Connecticut). Centrifugal extraction apparatus is available from Western States Machine Company (Hamilton, Ohio) and Silver-Weibull (Hasslehom, Sweden). Suitable membrane filtration systems are available from suppliers such as CeraMem Corp. (Waltham, Massachusetts), Koch Membrane Systems, Inc. (Wilmington, Massachusetts), and Osmonics, Inc. (Minnetonka, Minnesota).

[0071] The following table shows suitable characteristics for some of the process streams in Figure 1, namely RDS (weight % refractive dry substance), Purity (sucrose as a % of total solids), pH, and Temp.

Table 1

STREAM #	RDS	PURITY	pH	TEMP °F (°C)
38	12	85	6	100 (38)
45	12	85	8	160(71)
48	11	90	8	160 (71)
50	15	75	8	160(71)
58	8	20	8	160(71)
64	20	90	8	160 (71)
66	3	88	8	160(71)
72	1	50	6	100 (38)

[0072] Many variations of the process are possible. Suitable variations include reverse osmosis before ultrafiltration, sulfitation after ultrafiltration, and sterilization of the macerated beets by chemical or physical means. Separate treatment of the press juice 72 instead of returning it to the countercurrent vacuum filtration process is another alternative. It would also be possible to include treatment with some amount of lime and/or carbonation. However, it is presently preferred to operate the process without the use of either lime or carbonation.

[0073] Chromatographic separation could be used for further purification in this process. Chromatographic separation requires juice pretreatment and juice softening. Since the juice from the present process has been passed through membrane filtration and no lime has been added, it would be excellent feed to chromatographic separation.

[0074] Further use of membrane separation in the proposed process could allow for separation of sucrose from other beet juice components such as invert sugars and oligosaccharides.

[0075] It may be possible to reduce or eliminate chemicals used for pH adjustment and sulfitation when beets of superior quality are being processed. It is also possible to operate various unit operations at somewhat different process parameters than those specified in the above-described embodiment, or in the following examples.

[0076] Leaching of macerated beets has been demonstrated to be capable of achieving 99.8% recovery of sugars in six stages, each using fresh water. Ultrafiltration of juice has also been demonstrated to be capable of achieving 99.8% sugar recovery in six stages of diafiltration. However, this degree of extraction may be too ambitious for an industrial process since it involves excessive use of dilution water, which has to be removed eventually for recovery of sugar.

[0077] A mass balance of a process according to the present invention was prepared based on an input of 1,000 units of beets with 78% water, 17% RDS and 89% sucrose purity, and an assumed sugar recovery of about 99.5% in both extraction and diafiltration operations. Figure 2 shows a flow diagram of this embodiment of the process with the mass balance. The numbers in bold type are assumed based on experimental data and other available information. All other numbers are determined using constitutive and conservation relations. "EJ" refers to extracted juice, "UFP" refers to ultrafiltration permeate, "UFR" refers to ultrafiltration retentate, "DFP" refers to diafiltration permeate, "DFR" refers to diafiltration retentate, "MP" refers to mixed permeate, and "NSDS" refers to non-sugar dissolved solids.

[0078] In Figure 2 beets are macerated with juice from the second stage of the extractor. Macerated beets are fed to the first stage of the extractor and juice from this stage is fed to the ultrafiltration system. Pulp from the first stage moves through several stages of the extractor until nearly all the sugar (99.5%) is extracted. Fresh water is introduced in the last stage of the extractor. Extracted juice is processed by ultrafiltration to recover 90% of the juice as ultrafiltrate. The retentate is diafiltered five times its volume of fresh water. Combined ultrafiltration and diafiltration recover about 99.5% of the sugar in the feed.

[0079] There could be several improvements to the process of Figure 2. The wet pulp can be pressed to reduce moisture content to about 80% and the press water can be used to replace part of fresh water used in the extraction. Diafiltrate from the latter stages could also be used to replace some fresh water in the extraction process. These modifications would reduce the load on subsequent unit operations like drying or transport of pulp and reverse osmosis or evaporation of juice. However, these measures would reduce the efficiency of the extraction process, requiring more stages.

[0080] Although the process of the present invention can be operated in batch mode, it is especially well suited for continuous operation.

[0081] Another embodiment of the invention is shown in Figure 3. Beets received from the field are kept in a storage area 110. Fresh beets are typically used in the process, but frozen beets can also be used. Beets from the storage area are flumed in a conventional beet washing apparatus, in which dirt is removed from the exterior of the beets.

[0082] The washed beets are carried by conveying apparatus to macerating apparatus. The macerating apparatus can comprise, for example, one or more hammer mills 112 and one or more grinders 114. Suitable hammer mills can use a set of swinging blades mounted on a vertical shaft, which force the beet material through a discharge screen. Another suitable type of hammer mill uses fixed blades. Another suitable macerating apparatus comprises one or more attrition mills that use discs as the primary attrition device. The discs preferably have grooves therein to facilitate maceration, and the discs can be horizontal or vertical in positioning. It is also possible to use both disc mills and hammer mills in series (e.g., hammer mill followed by disc attrition mill) or to have only one type of mill, a hammer mill.

[0083] Partially macerated beets exiting the hammer mill 112 can be passed through the grinder 114, in which the beet material is further macerated. The macerated material leaving the grinder comprises pulp (i.e., fibrous material from the beets) and aqueous liquid that contains sucrose as well as other substances. Juice side streams 116 and 118 can be taken from the output of the hammer mill 112 and the grinder 114 and recycled into the feed to those devices. This increases the flow of liquid through the hammer mill 112 and the grinder 114 and helps carry along the solid portion of the beets.

[0084] The macerated material leaving the grinder 114 is passed through a separator 120 for separation of the liquid from the fibrous pulp. The separator 120 can suitably be a centrifuge, filter, or screen (e.g., a rotating or vibrating screen, or a Dorr-Oliver DSM screen), or a combination of two or more of these. In the embodiment of the process shown in Figure 3, the separator 120 comprises a centrifuge, which produces a pulp stream 122 and a juice stream 124. The pulp is passed through a series of screens 126 and 128, with a counter-current flow of aqueous liquid helping to remove residual sucrose from the pulp. These can be rotating, vibrating screens, or DSM screens. The counter-current flow is established by introducing a water stream 130 into a centrifuge 134 at the other end of the series of screens. The pulp stream 132 exiting the final screen 128 then passes into the centrifuge 134, in which it is separated into a low-water pulp material 136 and a recovered juice 138, with the latter being routed in counter-current flow to the pulp through the series of screens 128 and 126. This centrifuge 134 desweetens the pulp. A liquid 119 can be drawn from one of the screens, usually the first screen, and fed into the juice stream before the centrifuge 120. Optionally anti-foam can be added to the juice and fibre streams to reduce foaming.

[0085] The low water pulp 136 has some of its remaining water and sucrose removed in a press 140. The aqueous stream 142 from the press 140 is recycled into one of the screens 128. The residual fiber 144 that remains after pressing can be used as animal feed.

[0086] The juice stream 124 exiting the centrifuge 120 can optionally have an air stream 146 injected into it. This will oxidize color-forming materials in the juice (e.g., resulting in a color increase from 8,000 to 16,000), which aids in the formation of polymerised color bodies and thereby facilitates removal of the color bodies in the subsequent ultrafiltration. It is also possible to inject a stream 148 of hydrogen peroxide solution, in addition to or instead of injecting air. The hydrogen peroxide also assists oxidation and polymerisation of color-forming materials. Alternatively, ozone could be injected in place of hydrogen peroxide. The temperature of the juice is preferably increased at this point in the process by a heater 149, preferably to about 140-200 °F (60-93 °C), more preferably about 160-185 °F (71-85°C).

[0087] Optionally, the heated juice can be pre-filtered prior to the first ultrafiltration, in order to reduce its already low fiber content. The pre-filtration can be done, for example, with a rotating or vibrating screen 150. Preferably, the filter or screen 150 has a mesh size of about 30-100 microns, and removes the majority by weight of the fiber and silt remaining in the juice.

[0088] The heated and screened juice 152 can optionally have its pH adjusted by addition of a stream 154 that comprises, for example, aqueous sodium hydroxide, calcium hydroxide, or potassium carbonate. This pH adjustment helps prevent the inversion of sugars which can take place at elevated temperatures. Other chemicals may be also be used for pH adjustment, such as liquid potassium hydroxide or granular sodium or potassium carbonate. Preferably the pH of the juice after this step is between about 6.0-8.0, more preferably between about 6.5-7.5.

[0089] The juice after the pH adjustment, referred to herein as the ultrafiltration feed juice 156, is brought into contact with a first ultrafiltration membrane 158. This first ultrafiltration membrane is preferably tubular or spiral and preferably has a molecular weight cutoff of at least about 2,000 daltons and a pore size no greater than about 0.1 microns, more preferably having a molecular weight cutoff between about 4,000-500,000 daltons, most preferably between about 10,000-200,000.

[0090] The ultrafiltration step produces a first ultrafiltration permeate 160 and a first ultrafiltration retentate 162. In this embodiment of the process, the first ultrafiltration retentate 162 is then fed to a first ultrafiltration/diafiltration membrane 164 with addition of water 166. This ultrafiltration/diafiltration membrane can suitably have a pore size/molecular weight cutoff that is approximately the same as the first ultrafiltration membrane 158. This first diafiltration 164 produces a first diafiltration permeate 168 and a first diafiltration retentate 170 (also referred to as the molasses 1 stream). The diafiltration minimizes the amount of sucrose lost in the molasses (i.e., the concentration of sucrose on a dry solids basis (dsb) is lower in the retentate 170 than in the feed 162). It should be understood that there could be several stages of ultrafiltration 158 and/or diafiltration 164.

[0091] The first ultrafiltration permeate 160 typically will have a color of about 3,000-10,000 icu. The first ultrafiltration

permeate 160 and the first diafiltration permeate 168 are combined to form the feed 172 for a second ultrafiltration membrane 174.

[0092] Prior to the second ultrafiltration, a sulfitation stream 176 can be injected into the juice 172. This stream 176 can comprise, for example, sulfur dioxide, or sulfite, bisulfite, metabisulfite, or dithionite salts, such as aqueous ammonium bisulfite or sodium bisulfite (e.g., at about 35-65% concentration). Preferably, the residual level of sulfur dioxide in the juice after sulfitation is at least about 100 ppm. The sulfitation can take place at one or more points in the process, for example, at the time of slicing or macerating the beets, in the juice after it is separated from the pulp, in the feeds to the first or second ultrafiltrations or to the nanofiltration, and/or in the feed to the evaporator. Most preferably, the sulfitation is done in the feed to the second ultrafiltration. This sulfitation will prevent the color increase that can otherwise take place during membrane filtration and evaporation operations. Other antioxidants may also be used, as well as anti-foaming agents.

[0093] The second ultrafiltration membrane 174 preferably has a molecular weight cutoff of about 500-5,000, more preferably about 2,000-4,000. The second ultrafiltration produces a second ultrafiltration permeate 178 and a second ultrafiltration retentate 180. The retentate 180 is then mixed with second greens 234, the mother liquor recycled from the second white sugar crystallisation, and passed through a second ultrafiltration/diafiltration membrane 182 with added water 184. The membrane used for the second diafiltration can suitably have a pore size/molecular weight cutoff that is lower in pore size than the second ultrafiltration membrane 174. This is to remove raffinose and a membrane with a pore size in the range 500-1,000 daltons is preferred. This step produces a second diafiltration permeate 188, which is mixed with the second ultrafiltration permeate 178 and fed to a nanofilter 190, and a second diafiltration retentate 186 (also referred to as the molasses 2 stream). There could be more than one stage of membrane filtration in the second ultrafiltration 174 and/or the second diafiltration 182. The permeate 178 from the second ultrafiltration preferably will have color in the range of 1,500 - 3,500, or in some cases even less.

[0094] Optionally, the second diafiltration permeate 188 and/or the first diafiltration permeate 168 can be recycled into the diafiltration water streams.

[0095] Alternatively, or in addition to ultrafiltration/diafiltration, the retentate can be purified by a chromatographic separation in a simulated moving bed separator system. This is shown in Figure 4. This chromatographic separator 236 preferably is a multistage unit with from three to twenty stages, more preferably ten stages. It preferably has three product cuts, one being predominantly sucrose, stream 237, another being predominantly raffinose and ash, and the third being predominantly organic material including organic acids. The two non-sucrose streams can be mixed to give stream 186 (referred to as molasses 2). The resin used in the separator preferably is a strong acid cationic resin. The sucrose stream 237 is mixed with the feed to the evaporator. Alternatively, it could be added to the feed of the electrodialysis 192, or to the ion exchange 194, depending on the degree of removal of impurities.

[0096] The second ultrafiltration permeate 178 is then purified by nanofiltration, and optionally also ion exchange and/or electrodialysis, in any sequence. In the embodiment shown in Figure 3, the ultrafiltered juice 178 is first nanofiltered 190, followed by electrodialysis 192 and ion exchange softening 194. Although the sequence of these three operations can be varied, it is usually preferable to perform electrodialysis after nanofiltration.

[0097] The feed to the nanofiltration membrane typically comprises about 84 % sucrose, 3-6 % ash, and about 0.5-4.0 % invert sugar (all by weight on dsb). The nanofiltration membrane 190 separates the feed into a nanofiltration permeate 196 (also referred to as the molasses 3 stream) and a nanofiltration retentate 191 which will contain most of the sucrose from the beets. The nanofiltration permeate preferably contains at least about 30-60 % by weight of the ash (primarily Na, K, and Cl), at least about 30-50 % by weight of the invert (glucose and fructose), and at least about 25-50 % by weight of the betaine present in the nanofiltration feed 178. The nanofiltration will accomplish some color reduction from the nanofiltration feed to the retentate. A typical nanofiltration permeate will comprise 20 % sucrose, 25 % ash, 20 % invert, 8 % betaine and 25% other organics (dsb). Preferably, the nanofiltration retentate 191 will contain at least about 89-91 % by weight (dsb) sucrose and will have a concentration of about 15-28 Brix. Although nanofiltration can effectively remove potassium, it does not remove a large percentage of the citric, oxalic, and malic acid that is present.

[0098] The nanofiltration retentate 191 is then further purified by electrodialysis 192, which removes additional ash and various organic acids and other impurities, including some that cause undesirable color. Electrodialysis provides good removal of oxalic acid and malic acid, with the total ash removal typically being over 40%. The impurity stream 198 from the electrodialysis is combined with the streams 170, 186, and 196, to form a molasses product stream 200.

[0099] Although electrodialysis can achieve good removal of potassium, it does not typically remove a high percentage of the magnesium that is present. Therefore, the purified juice 193 from electrodialysis (which will typically contain about 92-94 % sucrose dsb) preferably is then softened by ion exchange unit 194 which contains at least one ion exchange resin. A strong cation exchange resin based on a gel or macro-porous matrix, with cross-linking ranging from 4 to 10%, is preferred. Examples of these are resins such as Rohm & Haas Amberlite IR120, or Purolite C 100. These will be used in the sodium or potassium form. The primary purpose of this step is to remove divalent cations, such as Ca and Mg, and replace them with monovalent cations, such as K and Na. This ion exchange step preferably removes at least about 95 % by weight of the Ca and Mg present.

[0100] The purified juice 202 from the ion exchange, which preferably comprises more than about 92 % sucrose (dsb), is then fed to one or more evaporators 204, in which a concentrated syrup 206 is formed (e.g., about 75 % dry solids) by removal of substantial quantities of water. Optionally, a sulfitation stream 205 can be injected into the evaporator. Preferably, the syrup will have a pH of about 6.5-7.5 and a temperature of about 160-180 °F (71-82 °C) during evaporation.

[0101] The concentrated syrup 206 is fed to a first crystallizer 208, in which water is boiled off and a first strike of white sugar crystals 210 is formed. The crystals 210 are centrifuged 212, washing with a water spray, to remove any residual liquid, and the remaining product is white sugar 214 (sucrose concentration of about 99.95%). The mother liquor 216 remaining after the first crystallization and centrifugation (typically containing about 84 - 88% sucrose dsb) is fed to a second crystallizer 218, in which a second strike of white sugar crystals 220 is formed. The crystals are also centrifuged 222 to produce white sugar 224. In prior art beet processes, the crystals produced in the second crystallizations were dissolved and recycled into the feed, because they were not pure enough to sell as white sugar. The present invention can achieve two strikes of highly pure white sugar, due to its improved purification capabilities. In a preferred embodiment, the crystallized sucrose (214 and 224) will comprise less than about 0.015 % by weight ash, more preferably less than about 0.01 % ash, and a color less than 35 iu.

[0102] The mother liquor 234 remaining after the second crystallization (also referred to as "greens" or "jets", and typically containing about 80% sucrose dsb) can be recycled, for example into the second ultrafiltration/diafiltration 182. Optionally, this greens recycle stream may be routed through a purification unit to remove raffinose. This purification can be done by chromatographic separation of raffinose (see Figure 4) (also resulting in dilution of the greens to about 60 Brix), or alternatively by enzymatic digestion of raffinose 228 (see Figure 3). Preferably, if this purification 228 is included in the process, the raffinose concentration in the greens is decreased to a level no greater than about 1.0 % dsb. The enzyme used to hydrolyse raffinose is α-galactosidase (melibiase), splitting raffinose into sucrose and galactose. This can be carried out in a batch fashion in a stirred tank reactor at 50°C.

[0103] The process of the present invention can include multiple stages of ultrafiltration, nanofiltration, diafiltration, ion exchange, and/or electrodialysis. For example, the first ultrafiltration shown in Figure 3 could take place in two or more stages of ultrafiltration, rather than taking place through a single membrane. Those skilled in the art will recognize that many other variations on the specific embodiment shown in the figure are also possible. It should also be recognized that the process can be operated at a variety of temperatures and other process conditions.

[0104] Additional embodiments of the invention are shown in Figures 5 and 6. Beets received from the field are kept in a storage area 110. Fresh beets are typically used in these embodiments of the process, but frozen beets can also be used. Beets from the storage area are flumed in a conventional beet washing apparatus, in which dirt is removed from the exterior of the beets. The washed beets can be sliced into cossettes (e.g., having a thickness of about ¼ inch (0.6 cm)) in a slicing apparatus 112.

[0105] The sliced beets are carried by conveying apparatus to a diffuser 114. Juice extraction is done by allowing the sugar to diffuse through the natural cell walls of beets. The cell walls allow sugars and other low molecular weight compounds to pass through but prevent the passage of high molecular weight compounds. This selective diffusion process has two advantages. Retaining the high molecular weight compounds helps produce a high purity juice. It also reduces filtration difficulties that are caused by polysaccharides and proteins that comprise the high molecular weight compounds. The solid material 122 remaining after diffusion is fed to a press apparatus 126, in which additional juice is recovered that can be recycled to the diffuser 114. The solids remaining after pressing are high in fiber and can be used as animal feed.

[0106] The rest of the processes shown in Figs. 5 and 6 are generally as described with respect to Figs. 3 and 4, respectively.

[0107] A variety of membrane configurations can be used in the present invention, including for example spiral, hollow fiber, and tubular membranes. These membranes can be made from a various materials including polymers, ceramics, carbon and sintered stainless steel. Membranes that have a negative surface charge are preferred since most compounds to be rejected are negatively charged.

[0108] Some of the equipment used in the process is conventional and well known to persons of ordinary skill in this field, such as beet washing equipment and evaporators. Beet macerating apparatus is commercially available from suppliers such as Bepex Reitz (Santa Rosa, California), Andriz Sprout Bauer (Philadelphia, Pennsylvania) and The Fitzpatrick Company (Elmhurst, Illinois). Beet diffusion apparatus is commercially available from suppliers such as BMA (Braunschweig, Germany) and Silver Engineering (Colorado Springs, Colorado). Centrifugal extraction apparatus is available from Dorr Oliver (Milford, Connecticut), Western States Machine Company (Hamilton, Ohio), and Silver-Weibull (Hasslehom, Sweden). Suitable membrane filtration systems are available from suppliers such as Koch Membrane Systems, Inc. (Wilmington, Massachusetts), Osmonics, Inc. (Minnetonka, Minnesota), PCI (UK), and SCT (France). Suitable ion exchange equipment and resins are available from Prosep (Roscoe, Illinois), IWT (Rockford, Illinois), Purolite (Philadelphia, Pennsylvania), and Dow Chemical (Midland, Michigan). Suitable electrodialysis equipment is available from Eurodia (Paris, France) and Ameridia (Somerset, New Jersey). Suitable chromatographic separation equipment is available from Prosep (Roscoe, Illinois) and Applexion (Paris, France). Suitable enzymes for digestion of raffinose

are available from Novo (Denmark) or Hokkaido Sugar Co (Japan).

[0109] It would also be possible to include in the process a treatment with some amount of lime and/or carbonation. However, it is presently preferred to operate the process without contacting the feed juice or any of the permeates with either lime or carbon dioxide in order to carry out carbonation. Lime or carbon dioxide can be added as bases.

Example 1

Expelled Juice Clarification

[0110] Macerated beet pulp was mixed with water and pressed in cloth bags to produce a sample of expelled juice. This sample was treated with sodium hydroxide, heated and used in a set of ultrafiltration trials. Two different spiral ultrafiltration membranes were used in the trial, a Hydranautics model NTR7410 membrane and a Koch model HFK131 membrane. The trials produced satisfactory flux rates, higher than comparable trials with conventional beet diffusion juice.

Table 2

Ultrafiltration of Expelled Juice - Trial Parameters and Fluxes						
Trial No.	Pretreatment	Membrane Type	Trial Conditions		Trial Results	
			Temp. °F (°C)	Pressure PSIG (kPa)	Recovery (%)	Flux LMH
1	NaOH-Heat	Spiral	150 (66)	70 (483)	86	30
2	NaOH-Heat	Spiral	150 (66)	70 (483)	86	25

[0111] There was a significant reduction in RDS and a very significant increase in sucrose purity across the membrane. Both membranes rejected over 99% of the color value. The increase in sucrose purity and color separation during these trials were much higher than comparable trials with conventional beet diffusion juice.

Table 3

Ultrafiltration of Expelled Juice - Separation Characteristics										
Trial No.	Recovery (%)	RDS (%)			Sucrose (% of RDS)			Color		
		Feed	Retn.	Perm.	Feed	Rem.	Perm.	Feed	Retn.	Perm.
1	86	8.9	10.0	7.7	85.8	78.4	91.1	67256	158785	925
2	86	8.9	10.0	7.8	85.8	78.4	90.6	67256	158785	1138
("Retn." refers to retentate and "Perm." to permeate.)										

Example 2

[0112] A beet maceration trial was conducted using a Bauer atmospheric disc refiner. This machine has two 12 inch (30 cm) discs with adjustable gap, one disc stationary and other disc driven by a 60 hp (45 kW) motor. About 20 kg of beets were used in the trial. Beets were chopped to 3/4 inch (1.9 cm) pieces to suit the screw feeder.

[0113] All the beet chips were passed through the machine in one pass. Water was used to push the material through the machine, which resulted in dilution of juice. A part of the macerated product was pressed in a bladder press at 20 psi (138 kPa) for about 15 minutes. Another part of the product was allowed to drain on a wire screen box.

Table 4

Material	Concentration
Juice from bladder press	9.2 Brix
Press cake from bladder press	32.5% dry solids
Filter cake from screen box	15.0% dry solids

[0114] The pulp from the first pass was processed through the machine again in a second pass. The gap between

the discs was set to about 10 mil (0.25 mm) for this pass. The macerated pulp was pressed in the bladder press at 20 psi (138 kPa) for about 15 minutes.

Table 5

Material	Concentration
Juice from bladder press	7.6 Brix
Press cake from bladder press	21.0% dry solids

[0115] (The lower solids content in the pass 2 bladder press cake was due to its higher thickness.)

[0116] Pass 2 pulp drained under vacuum had a dry solids content of 22%. When it was washed in excess water and drained under vacuum, the solids content was only 15%. This indicated that 2/3 of the solids in the pulp were dissolved and easily washable. The washed pulp had a residual sugar content of about 0.5%.

[0117] Pass 2 pulp had poor filtration characteristics when subjected to a vacuum on a filter paper. However, on a 0.5 mm screen, a 25 mm thick pulp layer had filtration rates around 5,000 gfd.

[0118] These studies produced the following results:

1. The disc refiner pulped the beet with low power consumption (~3kWh/ton).
2. The pulp had good vacuum filtration characteristics (~5,000 gfd (8,500 LMH) with 25 mm cake).
3. The vacuum filter cake (after washing) had low residual sugar (~0.5%).
4. The filter cake may be pressed to produce a drier pulp by-product (~30%).
5. The expelled juice had satisfactory ultrafiltration characteristics (25 gfd (43 LMH)).
6. Ultrafiltration rejected color bodies in the expelled juice well (99%).
7. The ultrafiltrate of expelled juice has good sugar boiling characteristics.

Example 3

[0119] About 3,000 lb (1361 kg) of beets were macerated in fixed hammer mills for about 30 minutes, producing about 400 gallons (1514 L) of juice. The maceration involved two passes. The first pass was through two grinders and two extractors, and the second pass was through one grinder and two extractors. The excess water added to the hammer mills to facilitate discharge of the macerated beets diluted the juice to about 4% RDS. The juice was filtered through a #200 mesh vibratory screen. No visible residue was left on the screen.

[0120] The juice was heated to about 170°F (77 °C) and ultrafiltered through a Koch HFK 131 ultrafiltration spiral membrane module with an 80 mil spacer. The inlet and outlet pressures were maintained at 60 and 40 psig (414-276 kPa). Table 6 summarizes the results.

Table 6

Ultrafiltration of Expelled Juice - Trial Parameters and Fluxes, and Separation Characteristics												
Time (min.)	Recovery (%)	Temp. °F(C°)	Flux (Imh)	RDS (%)			Sucrose (%) .			Color		
				Retn.	Perm.	Rej.	Retn.	Perm.	Rej.	Retn.	Perm.	Rej. (%)
0	0	176(80)	135	4.6	4.3	6.5	78.7	80.6	4.3	76,946	6,781	91.8
35	33	161 (72)	90	5.3	4.4	17.0	70.8	81.2	4.8	130,128	6,313	96.0
50	50	166(74)	90	6.4	4.6	28.1	61. 1	81.5	4.2	208,396	5,442	98.1
55	67	167 (75)	83	7.8	4.8	38.5	50.2	80.3	1.6	308.950	5,103	99.0
70	83	161 (72)	45	12.1	5.4	55.4	35.6	78.0	2.3	588,757	10,335	99.2
"Rej." refers to rejection. Note: This test was performed to evaluate the ability to process deteriorated beets. The feed beet material used for this test is substantially lower in purity than normal beets - this accounts for the lower permeate purities and higher permeate colors.												

Example 4

[0121] A set of leaching trials was conducted using a centrifuge as the leaching device. Macerated pulp was prepared by processing beets through a hammer mill of the Rietz Disintegrate type. The centrifuge was an American Machinery and Metals basket type centrifuge, whose basket was 18 inches (46 cm) in diameter and 10 inches (25 cm) deep, and was driven by a 3 hp (2 kW), 1,700 rpm electric motor. A sleeve made of filter cloth was used as a liner inside the basket to contain the filter cake.

[0122] A five-gallon volume of the macerated pulp was centrifuged for about two minutes and the extracted juice was collected. The cake was remixed with an equal volume of water and centrifuged again. This procedure was repeated six times. Samples of the extracted juice and cake were collected at the end of each run. The results of one trial are summarized in Table 7.

[0123] The results indicate that the sucrose content in the juice and pulp decreased by half in every step. This is to be expected since the cake was mixed with an equal volume of water at each step. The sugar content of the pulp after six steps was 0.03%. This translates to extraction of 99.8% of sugar in the beets.

Table 7

Leaching Trial Results							
Run #	Juice			Pulp			
	RDS %	Sucrose Purity (% of RDS)	% Sugar	Water %	RDS %	Sucrose Purity (% of RDS)	% Sugar
1	21.6	89.7	19.38	70.9	2.7	87.1	1.67
2	9.0	89.9	8.09	78.3	1.4	80.3	0.88
3	4.4	90.1	3.96	80.9	0.7	75.9	0.43
4	2.1	86.6	1.82	81.7	0.4	54.2	0.18
5	1.1	79.3	0.87	82.8	0.4	24.9	0.08
6	0.5	74.3	0.37	82.6	0.2	21.1	0.03

Example 5

[0124] A short trial was conducted with expelled juice ultrafiltrate/diafiltrate, to evaluate possibilities of preconcentration using reverse osmosis. The trial utilized a Hydranautics model ESPA spiral reverse osmosis membrane and was conducted at 800 psi (5516 kPa) at about 100 °F (38 °C). The flux and separation characteristics recorded in this trial are listed in Table 8.

Table 8

Reverse Osmosis of Extracted Juice Flux and Rejection Characteristics							
Recovery (%)	Flux (Lmh)	RDS (%)			Sucrose (% of RDS)		
		Retn.	Perm.	Rej.	Retn.	Perm.	Rej.
Feed		13.5			12.5		
10	65	14.4	0.4	97.2	13.4	0.3	97.5
60	31	25.2	1.4	94.4	23.2	1.3	94.5

Example 6a

[0125] Sliced beets (cossettes) were fed to a Rietz Disintegrator hammer mill at 420 kg/hour, and from there to an Andriz Sprout-Bauer grinder to provide a well macerated pulp. This fibre and juice was passed to the first centrifuge, a Mercone manufactured by Dorr-Oliver which was fitted with a 150 micron conical screen. The system was maintained at a temperature of 65 - 70 °C and the juice out of the first centrifuge was at 13-14 RDS.

[0126] The macerated beet fibre stream from the first centrifuge was fed to a system of 50 micron screens operating in a counter current fashion, and finally to a second Mercone centrifuge fitted with a 250 micron screen. This second

centrifuge desweetened the fibre providing a residual beet fibre contained 5.7% sugar and 88% water. Juice was recycled from the second centrifuge and counter-current through the system of screens to the first centrifuge at 3 - 5 gallons / minute (11-19 L/min). About 250 ppm on beets of antifoam oil (KABO 580) was added to the juice, while hot water was fed to the second centrifuge at 1 gallon / minute (4 L/min). The juice was adjusted to 70 °C, pH adjustment was carried out by adding sodium hydroxide solution to the maceration stage, and the final juice was pH trimmed as necessary. The final juice from the macerated beets was at 13.2 RDS (refractometric dry solids) and pH 6.4 (measured at ambient temperature). The apparent purity (Pol/Brix) was 82.6; conductivity ash 3.9% and colour 29,900 icu. It contained 0.6% fibre.

Example 6b

[0127] The juice from Example 6a, at about 70 °C, was fed to the first ultrafiltration. This was a PCI 12 foot (3.7 m) membrane module with tubular elements having a molecular weight cut off of 200 k Daltons, and a surface area of 2.7 m^2. The inlet pressure averaged 100 psi (689 kPa), the outlet 63 psi (434 kPa), and the cross flow rate was 537 litres / minute. The permeate flow rate was 2.6 litres / minute (corresponding to 60 Litres / square meter / hour). The permeate was 11.0 RDS; pH 6.5; apparent purity 83.8; colour 4705 icu, and ash 4.9%. The retentate was 11.1 RDS; 74.4 apparent purity; 5.1% ash and 60,800 icu colour. A similar membrane in series diafiltered the retentate with 1.0 litres / minute of water and delivered a further 1.9 litres / minute of permeate at 8 RDS.

Example 6c

[0128] The permeate from the first ultrafiltration system was fed at about 70 °C to a second ultrafiltration system which comprised two 4 inch (10 cm) spiral Osmonics GN membranes having a molecular weight cut off of 3,500 Daltons, with a surface area of 12 square meters. The inlet pressure averaged 65 psi (448 kPa), the outlet 50 psi (345 kPa). The total permeate flow rate averaged 1.4 litres / minute giving 7 Litres / square meter / hour. The permeate was 8.6 RDS; pH 7.0; apparent purity 83.6; colour 1666 icu, and conductivity ash 4.9% (5.4% sulphated ash). The retentate was 14.2 RDS; 83.3 apparent purity; conductivity ash 5% and 13,800 icu colour.

Example 6d

[0129] The permeate from the second ultrafiltration membrane was passed over a cation exchange resin (Purolite C155S) in the sodium form. The flow was 2.4 litres / minute and this was passed over 90 litres of resin at 1.6 Bed volumes / hour and at 70°C. The product was 7.1 RDS, at pH 6.4, the apparent purity was 83.5; colour 1505 icu, and conductivity ash 5.6% (5.2% sulphated ash). The ionic composition of the resin input and output streams was:

Table 9

	Ca	Mg	K	Na	Cl	PO4	SO4	Oxalate
Feed	0.003%	0.085%	0.97%	0.91%	0.49%	0.28%	0.091%	0.44%
Product	0.001%	0.038%	0.87%	1.000%	0.52%	0.30%	0.089%	0.37%

Example 6e

[0130] The resin product stream was treated by nano filtration with 3 stages of Desal 5DL membranes. The total membrane surface was 18 square metres, and the inlet pressure 450 psi (3013 kPa), and temperature 65 °C. The feed flow was 2.6 litres / min and the retentate flow 1.0 litres / min. Diafiltration water was introduced between the stages at 0.3 litres / min.

[0131] The retentate (product) stream was 25.4 RDS; 88.3 apparent purity; colour 1154 icu and 2.9% ash. The permeate was 1.8 RDS; 12.0 apparent purity; 3083 colour and about 20% ash.

[0132] By HPLC the composition of the streams (% on dry solids) was:

Table 10

	Sucrose	Glucose	Fructose	Raffinose	Betaine
Feed	82.9	0.22	0.75	0.52	1.83
Permeate	18.7	0.63	3.62	0.09	7.11

(continued)

	Sucrose	Glucose	Fructose	Raffinose	Betaine
Retentate	89.3	0.11	0.31	0.63	1.18

Example 6f

[0133] The product stream from the nano filtration was evaporated to give a syrup at 70.5 Brix. Approximately 1 ml / minute of ammonium bisulfite (ABS) solution was added to the feed juice to the evaporator. The ABS was a 65% stock solution diluted 1:1. The evaporator was a single effect APV plate and frame unit, and was operated at 8 psia (55 kPa absolute) and the syrup temperature was about 85°C. The feed flow rate was 1 litre / minute at 25.4 brix.

[0134] The liquor produced by the evaporator was crystallized under vacuum to give white sugar with colour 10.2 icu and a conductivity ash of 0.009%. The crystallisation was carried out in batch mode, in a crystalliser containing 50 litres of massescuite. The crystalliser was a pilot unit manufactured by Pignat of Genas, France. Crystallisation pressure and temperature were 20 in Hg abs (68 kPa absolute) and 70 - 75°C and crystallisation took 2 hours. The massecuite formed by crystallisation was centrifuged on a 2 foot basket centrifuge using a perforated basket. The mother syrup (separated by centrifugation) had an apparent purity of 81.8% and a colour of 2988 icu.

Example 7 (Alternative Ion Exchange Treatment)

[0135] A second ultrafiltration stage UF2 similar to that described in example 6c above but comprising two 4 inch (10 cm) spiral Osmonics GN (MWCO 3500 Daltons) and two 4 inch (10 cm) spiral Osmonics GM membrane (MWCO 3000 Daltons) modules was used to treat juice from a first stage of ultrafiltration. This juice was originally derived from macerated beets as described above. The modules ran at input pressures averaging 160 and 250 psi (1103 and 1724 kPa) respectively and delivered an overall 2.5 litres / min of permeate (pH 6.5, 9 RDS and 1890 colour). The permeate was treated with a cation exchange resin (Purolite PCR) in the potassium form at 70°C and 0.9 Bed Volumes / hour. The product was at pH 7.0, 8.9 RDS, 85.1 purity and 1959 colour. The input and output compositions (% on dry solids) were:

Table 11

	Ca	Mg	K	Na	Ash
Resin feed	0.006%	0.158%	1.14%	0.67%	5.4%
Resin product	0.006%	0.003%	3.39%	0.06%	7.8%

Example 8 (Electrodialysis)

[0136] The product from the ion exchange resin in the potassium form in Example 7 was treated by nano filtration (operated as in Example 6e above). The product (retentate) was 23 RDS, 89.5% purity (by HPLC), 4.5% as sulphated ash and 1800 icu colour. It was treated by electrodialysis in a stack comprising 40 cationic/anionic membrane pairs; each pair had 0.1 m^2 of membrane surface. The membranes used were manufactured by Tokuyama Corp of Japan, the anion membrane was AE506sb, and the cation membrane was CMXsb. The stack operated at 45 - 55 °C; with 18 - 30 volts and a current of 2 - 3 amps. The anolyte and catholyte systems contained dilute sulphamic acid (20 mS/cm conductivity) which circulated through the stack at 3 gpm (11 L/min). The stream being treated circulated at 8 - 10 gpm (30-38 L/min), and flowed through the whole system at about 1 litre / minute. The concentrate stream was 0.5% sodium chloride solution. The electrodialysed product stream was at 94.4% purity (by HPLC) and had 2.2% sulphated ash (calculated from the cation composition as shown below where the ionic composition of the feed and product streams is expressed as % ions on solids.

Table 12

	Ca	Mg	K	Na	Ash
Feed	0.002%	0.009%	1.86%	0.104%	4.5%
Product	0.001%	0.003%	0.88%	0.079%	2.2%

[0137] The product from this experiment was evaporated and crystallised to white sugar as in Example 6f. On evaporation (in the presence of ammonium bisulphite sufficient to give the 280 ppm of residual SO_2) the product was at 70

RDS and 1700 icu colour. Crystallisation under vacuum yielded a white sugar with a colour of 17 icu and a conductivity ash of 0.007%. The mother syrup (separated by centrifugation) had an apparent purity of 84% and 4560 icu colour.

Example 9

[0138] Juice from the beet maceration system, as described in Example 6a, was filtered in a spiral ultrafiltration membrane. It was first pre-filtered through a 200 micron bag filter to remove any fibres that had passed through the centrifuge screen. The membrane used was a 4 inch (10 cm) Osmonics JY spiral having a membrane area of 4.8 square meters and a cut off of 100,000D. The temperature of the juice was 140°F (60°C) and the inlet and outlet pressures were 50 psi and 30 psi (345 and 207 kPa) respectively. The feed rate was 3.6 litres per minute, and permeate and retentate flows were both 1.8 litres per minute at 13 and 14.2 RDS respectively, giving a flux rate of 22.5 litres / square meter / hour at a concentration factor of x2. The feed colour was 1232 iu and the permeate colour was 6475 iu. This juice can be further processed to white sugar using steps 6c, 6d, 6e, and 6f.

Example 10

[0139] The juice comprising a mixture of the mother liquor from white sugar crystallisations and the retentate from a second ultrafiltration can be evaporated to 60 RDS and passed at a rate of 0.9 litres/hour over a simulated moving bed separating system, containing 5.8 litres of resin distributed among 10 cells. Water can be injected at 4 litres per hour and the system operated at a temperature of 70 °C. Three fractions can be collected from the system containing respectively, most of the organics; most of the sucrose; and most of the raffinose plus other organic materials. Typical properties of each of these fractions are given in the tables below. ("Organics" represent materials calculated by difference from the analytical results.)

Table 13

	Flow l/hour	RDS	Sucrose	Invert	Ash	Raffinose	Organics	Colour iu
Feed	0.9	60	66.6	4.0	5.9	8.2	15.3	33,000
Organic fraction	0.7	1.1	13.2	9.8	16	0.0	61	29,700
Sucrose fraction	1.84	29.3	96.0	0.7	0.2	3.1	0.0	6090
Raffinose fraction	2.5	8.4	13.7	9.8	16.6	20.4	39.5	90,300

[0140] The sucrose fraction obtained can be 96% pure and represent a recovery of 92.5% of the sucrose input.

Example 11

[0141] 450 gms of the mother syrup from the first crystallisation of white sugar (at about 75 RDS, and containing 2.3% raffinose on solids) was diluted to 30 RDS with water. The pH was adjusted to 5.0 by adding dilute sulphuric acid and the solution temperature brought to 50 °C. 2.5 x 10^7 units of pelleted α-galactosidase enzyme, were added (12.2 gm) and the solution stirred at 50°C for 2 hours. The resulting juice contained 0.7% raffinose on solids.

Example 12

[0142] The mother syrup from the crystallisation of a first strike of white sugar (colour 3147 icu at 83.1% apparent purity) was crystallised under vacuum to give a second crop of white sugar with colour 20 icu and a conductivity ash of 0.01%. The crystallisation was carried out in batch mode in a crystalliser containing 50 litres of massecuite. The crystalliser was a pilot unit manufactured by Pignat of Genas, France. Crystallisation pressure and temperature were 20 inch Hg abs and 70 - 75 °C, and crystallisation took about 2 hours. The massecuite formed by crystallisation was centrifuged on a 2 foot (0.6 m) basket centrifuge using a perforated basket. The mother syrup (separated by centrifugation) had an apparent purity of 81.1% and a colour of 4155 icu.

Example 13a

[0143] Beet juice from a factory diffuser (colour about 4200 icu) was fed to the first centrifuge (with a 150 micron screen) to remove residual fibres, and heated to 70 °C. It was aerated vigorously, screened (50 micron) and adjusted with sodium hydroxide to pH 8.0. The resulting juice was 17.5 RDS, apparent purity 85.1, colour 16400 and conductivity

ash 5.3. It contained 0.09% fibre.

[0144] This juice was fed at 73 °C to the first ultrafiltration: an Osmonics PW, 4 inch (10 cm) diameter membrane module with spiral elements having a molecular weight cut off of 10 - 15,000 Daltons, and a surface area of 4.3 m^2. The inlet pressure averaged 66 psi (455 kPa), the outlet 45 psi (310 kPa), and the cross flow rate was 193 litres / minute. The permeate flow rate was 1.9 litres / minute (corresponding to 26 litres / square meter / hour). The permeate was 15.5 RDS; apparent purity 85.6; colour 6697 icu, and ash 5.3%. The retentate was 12.5 RDS and 83.4 apparent purity. Diafiltration of the retentate using a PCI 20,000 Dalton molecular weight cut off tubular membrane produced a further 1.6 litres / min of permeate at 7 RDS. The permeates were mixed.

Example 13b

[0145] The combined permeates from the first ultrafiltration system were fed at 65 °C to a second ultrafiltration system using two Osmonics GK and two Osmonics GE membrane 4 inch (10 cm) spiral modules. These membranes have 2000 Dalton and 1000 Dalton molecular weight cut offs respectively. It ran at input pressures averaging 250 psi (1724 kPa) and delivered a total of 2.2 litres / min of permeate (13.5 RDS, 85.0 apparent purity, 6.5% conductivity ash and 3297 colour). The retentate was 21.4 RDS and 83.8 apparent purity. The total membrane area was 24 square meters and the average flux rate was 5.5 litres/square meter /hour

Example 13c

[0146] The permeate from the second ultrafiltration membrane system was treated by nano filtration with 2 stages of 4 inch (10 cm) diameter Desal DS5 membranes. The total membrane surface was 12 square metres, the inlet pressure 450 psi (3103 kPa), and temperature 150 °F (66 °C). The feed flow was 0.75 gpm (2.5 L/min) and the retentate flow 0.3 gpm (1 L/min). Diafiltration water was introduced into the second stage at 2.75 gph. The retentate (product) stream was 24.9 RDS; 90.6 apparent purity (90.8% by HPLC); colour 2802 icu and 4.2% conductivity ash. The permeate was 5.2 RDS and 38.3 apparent purity.

Example 13d

[0147] The nano filtration retentate from Example 13c was treated by electrodialysis in a stack comprising 40 cationic/anionic membrane pairs, each pair had 0.1 m^2 of membrane surface. The stack operated at 45 - 55 °C; with 18 - 30 volts and a current of 2 - 3 amps. The anolyte and catholyte systems contained dilute sulphamic acid (20 mS/cm conductivity) which circulated through the stack at 3 gpm (11 L/min). The stream being treated circulated at 8 gpm (30 L/min), and the equipment was operated in a batch mode. The stack was operated at 40 - 50 °C; with 12 - 23 volts and a current of 3 amps. The feed and product ionic compositions were:

Table 14

	% of Ions on Solids								% Ash
	Ca	Mg	K	Na	Cl	PO4	SO4	Oxalate	
Feed	0.001	0.12	0.65	0.61	0.049	0.146	0.523	0.628	3.9
Product	0.003	0.02	0.19	0.19	0.021	0.046	0.061	0.073	1.2

[0148] The product was at 92.5% purity (by HPLC)

Example 13e

[0149] The product from electrodialysis (after adding about 900 ppm of SO_2 as an ammonium bisulphite solution) was evaporated to give a syrup at 69 Brix (colour 3060 icu). The evaporator was a single effect APV plate and frame unit, and was operated at 8 psia (55 kPa abs) and the syrup temperature was about 85°C. The feed into the evaporator was 1 litre / minute at 24 Bx.

[0150] The evaporated syrup was crystallised under vacuum to give a white sugar with 17.3 icu colour and a conductivity ash of 0.007%. The crystallisation was carried out in batch mode in a crystalliser containing 50 litres of massecuite. The crystalliser was a pilot unit made by Pignat of Genas, France. Crystallisation pressure and temperature were 20 in Hg abs (68 kPa abs) and 70 - 75°C and crystallisation took 2 hours. The massecuite formed by crystallisation was centrifuged on a 2 foot (0.6 m) basket centrifuge using a perforated basket.

[0151] The mother syrup (separated by centrifugation) had an apparent purity of 80.3% and a colour of 5380 icu.

Example 13f

[0152] The mother syrup produced by the methods of Example 13e was further crystallised in the Pignat crystalliser at 20 in Hg abs (68 kPa) and 70 - 75°C over 3 hours. The massecuite was centrifuged on a 2 foot (0.6 m) basket centrifuge and gave a second sugar of color 40 iu and 0.019 ash.

Example 14a

[0153] 55 gallons (208 L) of fresh beet diffuser juice at 16.5 Brix and 3850 icu colour, was adjusted to pH 8 with sodium hydroxide solution. 2.2 litres of 3% (v/v) hydrogen peroxide were added (0.03% on juice or 0.19% on solids). The juice heated to 80 °C for 60 min during which time the colour rose to 14,000 icu. Ultrafiltration through a 4000 Dalton molecular weight cut off PCI tubular membrane running at 300 psi (2068 kPa) yielded a permeate at 2100 colour and a retentate at 50,000 icu.

[0154] The permeate was evaporated and crystallised under vacuum, in equipment and under conditions similar to those in Example 13e. Colour was generated during this evaporation to give a crystallisation massecuite at 4450 icu. Centrifugation and washing gave crystals at 46 icu. In a further experiment, 200 ppm $S0_2$ was added to the crystallisation giving 34 icu color crystals, from a massecuite of 3950 colour.

Example 14b

[0155] The work in Example 14a was repeated, but feeding the oxidation with beet diffuser juice that had been heated and aerated as described in Example 13a. The feed colour was 12,123 icu. Peroxide treatment raised this to 14,473 icu and ultrafiltration gave a permeate at 2707 icu.

Example 15

[0156] The juice comprising a mixture of the mother liquor from white sugar crystallisations and the retentate from a second ultrafiltration can be evaporated to 60 RDS and passed at a rate of 1.0 litres/hour over a simulated moving bed separating system, containing 5.8 litres of resin distributed among 10 cells. Water can be injected at 4 litres per hour and the system operated at a temperature of 70 °C. Three fractions were collected from the system containing respectively, most of the organics; most of the sucrose; and most of the raffinose plus other organic materials. Typical properties of each of these fractions are given in the tables below. ("Organics" represent materials calculated by difference from the analytical results.)

Table 15

	Flow l/hour	RDS	Sucrose	Invert	Ash	Raffinose	Organics	Colour iu
Feed	1.0	60	67.5	5.0	6.2	7.4	17.3	37,000
Organic fraction	0.7	1.0	12	11.1	18.2	0.0	58	31,200
Sucrose fraction	1.9	27.9	96.5	1.0	0.4	2.5	0.0	6900
Raffinose fraction	2.6	9.1	15.1	8.7	19.2	22.3	42.5	85,700

[0157] The sucrose fraction obtained typically is 96.5% pure and represents a recovery of 90.5% of the sucrose input.

Example 16

[0158] 500 grammes of the mother liquor from the first crystallisation of white sugar (at about 75 RDS, and containing 2.6% raffinose on solids) was diluted to 30 RDS with water. The pH was adjusted to 5.0 by adding dilute sulphuric acid and the solution temperature brought to 50 °C. 2.5×10^7 units of pelleted alpha galactosidase enzyme were added (12.2 grammes) and the solution stirred at 50 °C for 2 hours. The resulting juice was analysed and found to contain 0.9% raffinose on solids.

Example 17

[0159] The mother syrup from the crystallisation of a first strike of white sugar (colour 4094 icu at 80.8% apparent purity) was crystallised under vacuum to give a second crop of white sugar with colour 28 icu and a conductivity ash of 0.024%. The crystallisation was carried out in batch mode in a crystalliser containing 50 litres of massecuite. The crystalliser was a pilot unit manufactured by Pignat of Genas, France. Crystallisation pressure and temperature were 20 inch Hg abs (68 kPa abs) and 70 - 75 °C, and crystallisation took about 2 hours. The massecuite formed by crystallisation was centrifuged on a 2 foot (0.6 m) basket centrifuge using a perforated basket. The mother syrup (separated by centrifugation) had an apparent purity of 77.4% and a colour of 5807 icu.

[0160] The preceding description of specific embodiments of the present invention is not intended to be a complete list of every possible embodiment of the invention. Persons skilled in this field will recognize that modifications can be made to the specific embodiments described here that would be within the scope of the present invention.

Claims

1. A process for producing sugar from beets, comprising the steps of:

(a) macerating sugar beets or pieces thereof, thereby producing a macerated material that comprises pulp and liquid,
(b) separating the liquid in the macerated material from the pulp at a temperature of at least about 65° C to produce a sucrose-containing feed juice;
(c) filtering the sucrose-containing feed juice through a first ultrafiltration membrane that has a first molecular weight cutoff, thereby producing a first ultrafiltration permeate and a first ultrafiltration retentate;
(d) filtering the first ultrafiltration permeate through a second ultrafiltration membrane that has a second molecular weight cutoff that is lower than the first molecular weight cutoff, thereby producing a second ultrafiltration permeate and a second ultrafiltration retentate; and
(e) filtering the second ultrafiltration permeate through a nanofiltration membrane, thereby producing a nanofiltration permeate and a nanofiltration retentate, wherein the nanofiltration retentate has a higher concentration of sucrose on a dry solids basis than the feed juice in step (c).

2. The process of claim 1, further comprising the step of purifying either the second ultrafiltration permeate or the nanofiltration retentate by at least one method selected from the group consisting of ion exchange and electrodialysis.

3. The process of claim 2, wherein the nanofiltration retentate is purified by electrodialysis, thereby producing an electrodialyzed juice and an electrodialysis residue.

4. The process of claim 3, wherein the electrodialyzed juice is softened by ion exchange, thereby producing a softened purified juice.

5. The process of claim 4, wherein the nanofiltration electrodialysis, and ion exchange remove at least about 65% by weight of the Ca, Mg, K, Na and their associated inorganic and organic anions that are present in the second ultrafiltration permeate.

6. The process of claim 3, wherein at least two of the first ultrafiltration retentate, the second ultrafiltration retentate, the nanofiltration permeate and the electrodialysis concentrate or residue are combined to produce molasses.

7. The process of claim 4, further comprising evaporating the purified juice to produce a concentrated syrup, and crystallizing white sugar from the concentrated syrup.

8. The process of claim 7, wherein the purified juice has an ash concentration of no greater than about 2.5% by weight on a dry solids basis.

9. The process of claim 8, wherein the purified juice has an ash concentration of no greater than about 2.0% by weight on a dry solids basis.

10. The process of claim 9, wherein the purified juice has an ash concentration of no greater than about 1.0% by weight on a dry solids basis.

11. The process of claim 7, wherein the process comprises two crystallizations of white sugar from the concentrated syrup.

12. The process of claim 7, wherein a mother liquor remains after crystallization of white sugar from the concentrated syrup, and the mother liquor is recycled to one of the ultrafiltration membranes.

13. The process of claim 4, wherein at least one aqueous stream selected from the group consisting of the feed juice, the first ultrafiltration permeate, the second ultrafiltration permeate, the nanofiltration retentate, and the purified juice is contacted with an agent selected from the group consisting of sulfur dioxide, sulfite salts, bisulfite salts, metabisulfite salts, dithionite salts, and mixtures thereof, in an amount sufficient to provide an equivalent concentration of sulfur dioxide in the stream of at least about 100 ppm.

14. The process of claim 1, wherein air is introduced into the feed juice prior to the first ultrafiltration to polymerise color bodies.

15. The process of claim 1, wherein hydrogen peroxide, ozone, or a combination thereof is introduced into the feed juice prior to the first ultrafiltration.

16. The process of claim 1, wherein the pH of the juice is adjusted to about 6-8 by addition of a base, prior to the first ultrafiltration.

17. The process of claim 1, wherein no more than about 5% of the sucrose present in the beets remains in the pulp after liquid is separated therefrom.

18. The process of claim 17, wherein no more than about 3% of the sucrose present in the beets remains in the pulp after liquid is separated therefrom.

19. The process of claim 1, further comprising the step of removing residual beet fibers and silt from the separated liquid, by at least one method selected from the group consisting of screening and filtration, prior to the first ultrafiltration.

20. The process of claim 19, wherein the screening or filtration removes at least 90% by weight of all fibers and silt having a largest dimension of about 150 μm or greater.

21. The process of claim 20, wherein the screening or filtration removes at least 90% by weight of all fibers and silt having a largest dimension of about 50 μm or greater.

22. The process of claim 1, wherein the beets are macerated by passing the beets through a hammer mill, whereby the beets are converted into a mixture of pulp and sucrose-containing liquid.

23. The process of claim 22, wherein the macerated beets are also passed through a grinder.

24. The process of claim 22, wherein liquid is separated from the pulp by centrifugation.

25. The process of claim 1, wherein the first ultrafiltration retentate is diafiltered through at least a first diafiltration/ultrafiltration membrane, thereby producing a first diafiltration permeate and a first diafiltration retentate; and wherein the first diafiltration permeate is filtered through the second ultrafiltration membrane.

26. The process of claim 25, wherein the second ultrafiltration retentate is diafiltered through at least a second diafiltration/ultrafiltration membrane, thereby producing a second diafiltration permeate and a second diafiltration retentate; and wherein the second diafiltration permeate is filtered through the nanofiltration membrane.

27. The process of claim 26, wherein at least the first diafiltration retentate, the second diafiltration retentate, and the nanofiltration permeate are combined to produce molasses.

28. The process of claim 1, further comprising evaporating the nanofiltration retentate to produce a concentrated syrup, and crystallizing white sugar from the concentrated syrup.

29. The process of claim 28, wherein a mother liquor remains after crystallization of white sugar from the concentrated syrup, and the mother liquor is recycled to one of the ultrafiltration membranes.

30. The process of claim 1, wherein the feed juice is at a temperature of about 140-200° F during filtration through the first ultrafiltration membrane.

31. The process of claim 30, wherein the feed juice is at a temperature of about 160-185° F during filtration through the first ultrafiltration membrane.

32. The process of claim 1, wherein the first ultrafiltration membrane has a molecular weight cutoff of at least about 2,000 daltons and a pore size no greater than about 0.1 microns.

33. The process of claim 32, wherein the first ultrafiltration membrane has a molecular weight cutoff of about 4,000-200,000 daltons.

34. The process of claim 1, wherein the first ultrafiltration permeate has a color of about 3,000-10,000 icu.

35. The process of claim 1, wherein the second ultrafiltration membrane has a molecular weight cutoff of about 500-5,000 daltons.

36. The process of claim 35, wherein the second ultrafiltration membrane has a molecular weight cutoff of about 2,000-4,000 daltons.

37. The process of claim 1, wherein the second ultrafiltration permeate has a color no greater than about 4,000 icu.

38. The process of claim 1, wherein the second ultrafiltration permeate has a color no greater than about 2,500 icu.

39. The process of claim 1, wherein the nanofiltration permeate comprises at least about 30% by weight on a dry solids basis of the ash present in the feed juice.

40. The process of claim 1, wherein the nanofiltration permeate comprises at least about 30% by weight on a dry solids basis of the invert sugars present in the feed juice.

41. The process of claim 1, wherein the nanofiltration permeate comprises at least about 25% by weight on a dry solids basis of the betaine present in the feed juice.

42. The process of claim 1, wherein at least one aqueous stream selected from the group consisting of the feed juice, the first ultrafiltration permeate, the second ultrafiltration permeate, and the nanofiltration retentate is contacted with an agent selected from the group consisting of sulfur dioxide, sulfite salts, bisulfite salts, metabisulfite salts, dithionite salts, and mixtures thereof, in an amount sufficient to provide an equivalent concentration of sulfur dioxide in the stream of at least about 100 ppm.

43. The process of claim 1, where no lime and no carbon dioxide are contacted with any of the permeates.

44. A process for producing sugar from beets, comprising the steps of:

(a) macerating sugar beets or pieces thereof, thereby forming pulp that comprises sucrose-containing aqueous liquid;
(b) separating the sucrose-containing liquid from the pulp at a temperature of at least about 65° C;
(c) filtering the sucrose-containing liquid through a first ultrafiltration membrane that has a molecular weight cutoff of about 4,000-200,000 daltons, thereby producing a first ultrafiltration permeate that has a color no greater than about 10,000 icu and a first ultrafiltration retentate;
(d) filtering the first ultrafiltration permeate through a second ultrafiltration membrane that has a molecular weight cutoff of about 2,000-4,000 daltons, thereby producing a second ultrafiltration permeate that has a color no greater than about 4,000 icu and a second ultrafiltration retentate;
(e) filtering the second ultrafiltration permeate through a nanofiltration membrane; thereby producing a nanofiltration permeate and a nanofiltration retentate, wherein the nanofiltration retentate has a higher concentration of sucrose on a dry solids basis than the sucrose-containing liquid in step (b);

(f) purifying the nanofiltration retentate by at least one method selected from the group consisting of ion exchange and electrodialysis, thereby producing an evaporator feed;
(g) evaporating water from the evaporator feed to produce a concentrated syrup; and
(h) crystallizing white sugar from the concentrated syrup.

45. The process of claim 44, wherein the process comprises at least two crystallizations of white sugar from the concentrated syrup.

46. The process of claim 44, wherein a mother liquor produced in the crystallization comprises raffinose, and at least 75% by weight of the raffinose is removed from the mother liquor in a simulated moving bed chromatographic separator, and the treated liquor is recycled.

47. The process of claim 46, wherein the recycled liquor is subjected to further purification, evaporation and crystallisation.

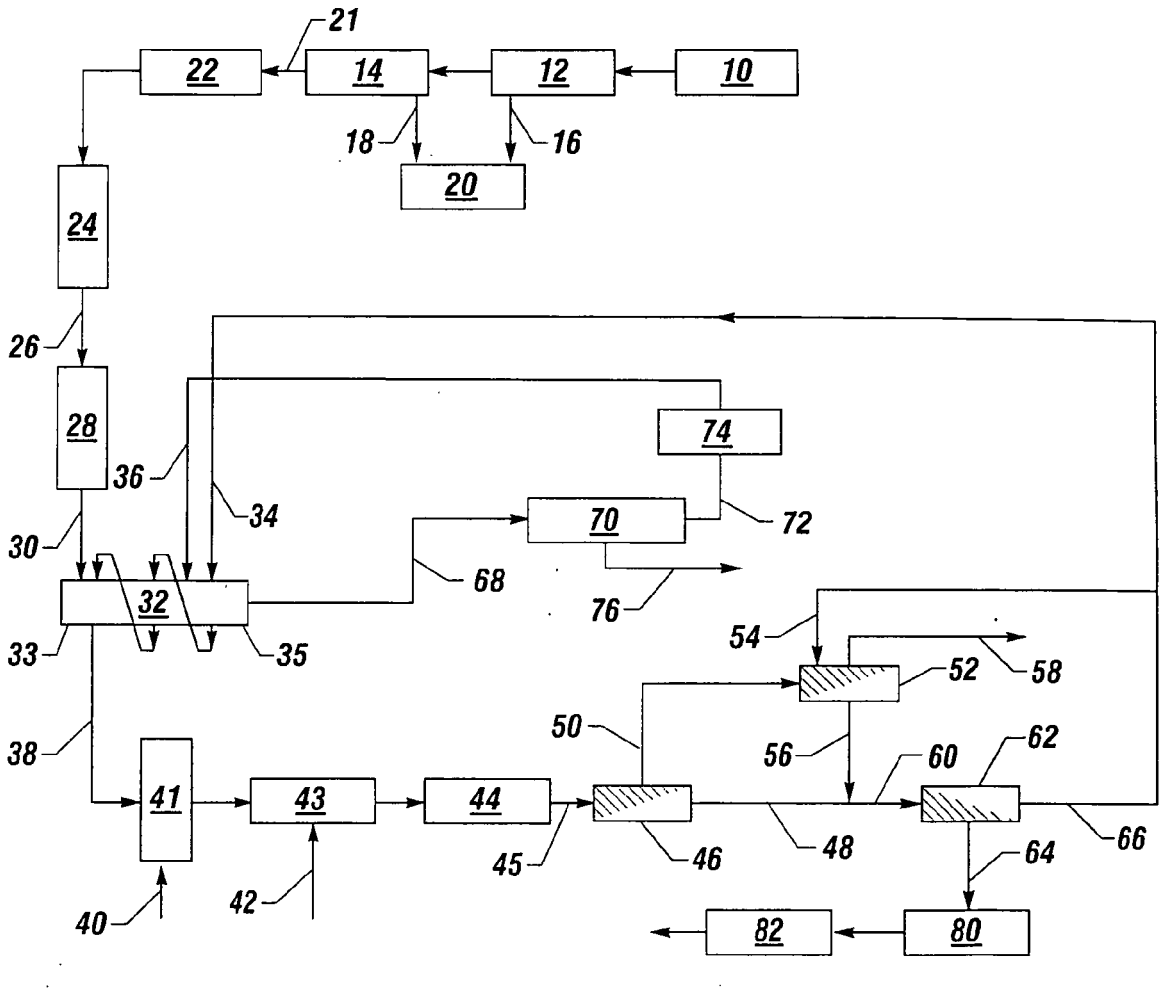
48. The process of claim 44, wherein a mother liquor produced in the crystallization comprises raffinose, and at least 75% by weight of the raffinose is removed from the mother liquor using melibiase enzyme, and the treated liquor is recycled to the feed of the second ultrafiltration membrane.

49. A process for purifying a sucrose-containing juice obtained from sugar beets, comprising the steps of:

(a) macerating sugar beets or pieces thereof, thereby forming pulp that comprises sucrose-containing aqueous liquid;
(b) separating the sucrose-containing liquid from the pulp at a temperature of at least about 65° C;
(c) introducing sufficient air into the juice to cause polymerisation of color bodies;
(d) heating the juice to a temperature of about 140-200° F; and
(e) removing at least some of the colour bodies from the juice by membrane filtration through at least one ultrafiltration membrane or nanofiltration membrane.

50. The process of claim 49, wherein the membrane filtration comprises:

filtering the juice through a first ultrafiltration membrane that has a first molecular weight cutoff, thereby producing a first ultrafiltration permeate and a first ultrafiltration retentate;
filtering the first ultrafiltration permeate through a second ultrafiltration membrane that has a second molecular weight cutoff that is lower than the first molecular weight cutoff; thereby producing a second ultrafiltration permeate and
a second ultrafiltration retentate; and
filtering the second ultrafiltration permeate through a nanofiltration membrane; thereby producing a nanofiltration permeate and a nanofiltration retentate.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82
80

FIG. 1

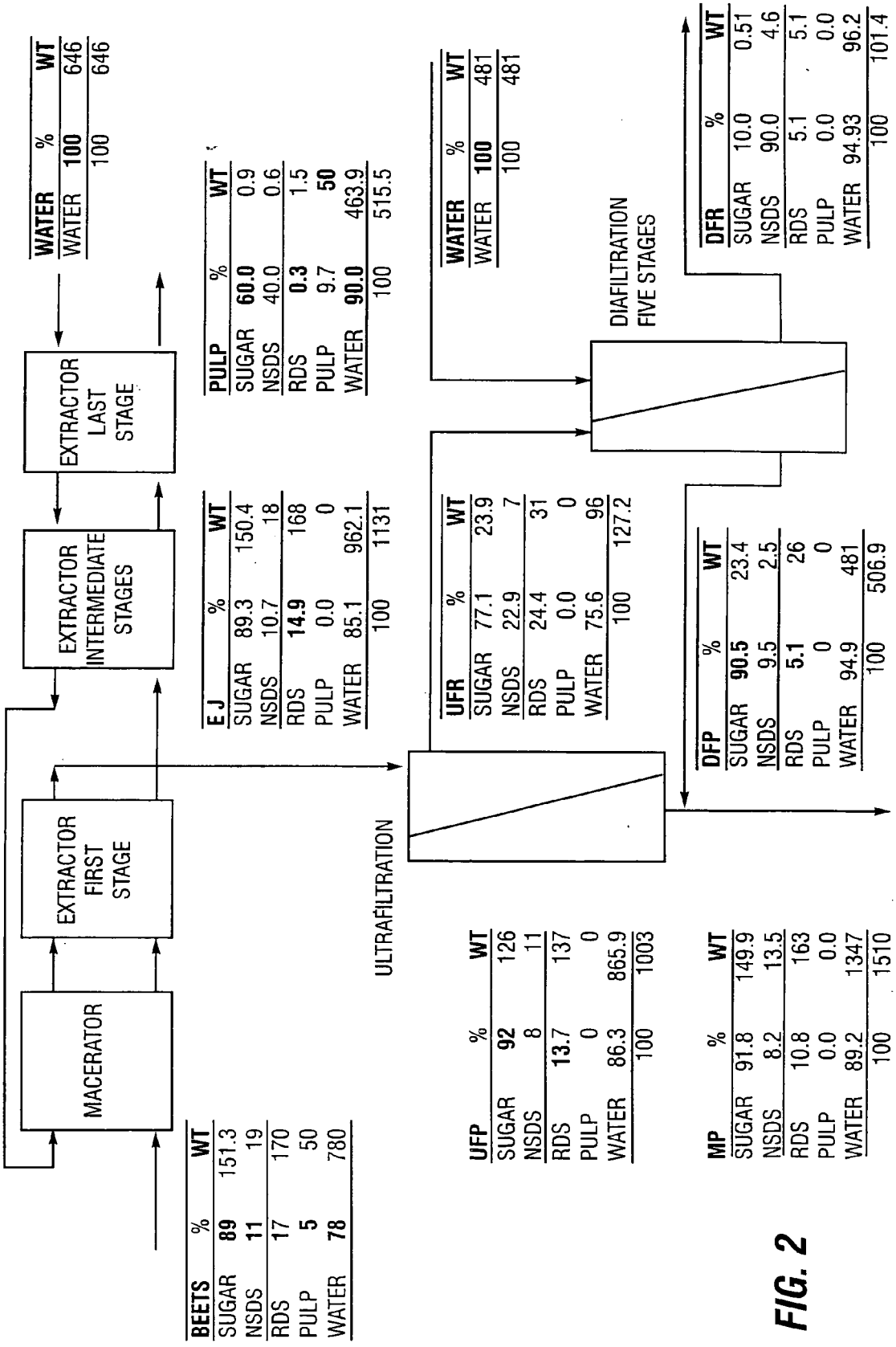
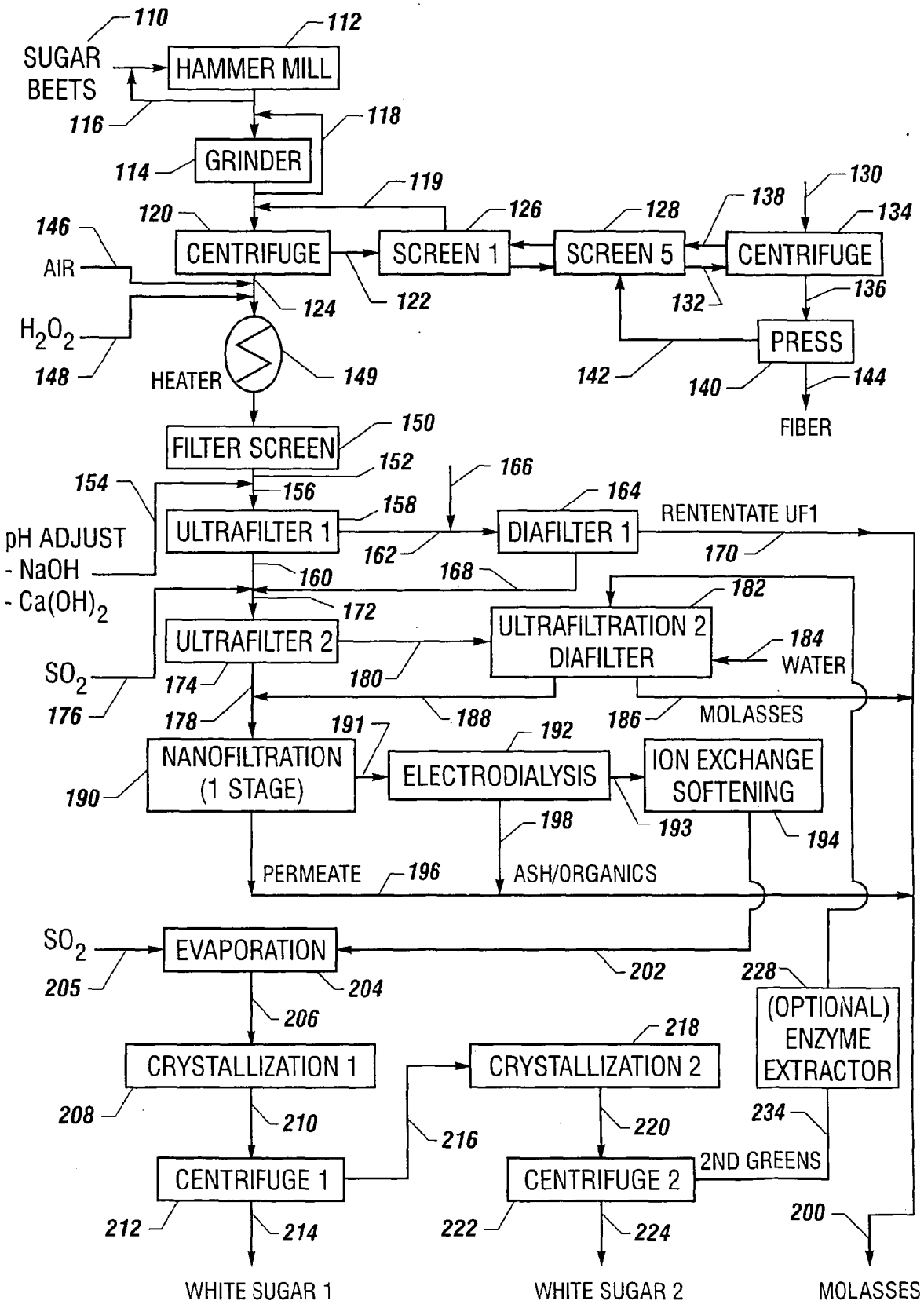

MACERATOR
EXTRACTOR FIRST STAGE
EXTRACTOR INTERMEDIATE STAGES
EXTRACTOR LAST STAGE
ULTRAFILTRATION
DIAFILTRATION FIVE STAGES
BEETS % WT
SUGAR 89 151.3
NSDS 11 19
RDS 17 170
PULP 5 50
WATER 78 780
WATER % WT
WATER 100 646
100 646
EJ % WT
SUGAR 89.3 150.4
NSDS 10.7 18
RDS 14.9 168
PULP 0.0 0
WATER 85.1 962.1
100 1131
PULP % WT
SUGAR 60.0 0.9
NSDS 40.0 0.6
RDS 0.3 1.5
PULP 9.7 50
WATER 90.0 463.9
100 515.5
UFR % WT
SUGAR 77.1 23.9
NSDS 22.9 7
RDS 24.4 31
PULP 0.0 0
WATER 75.6 96
100 127.2
WATER % WT
WATER 100 481
100 481
UFP % WT
SUGAR 92 126
NSDS 8 11
RDS 13.7 137
PULP 0 0
WATER 86.3 865.9
100 1003
DFP % WT
SUGAR 90.5 23.4
NSDS 9.5 2.5
RDS 5.1 26
PULP 0 0
WATER 94.9 481
100 506.9
DFR % WT
SUGAR 10.0 0.51
NSDS 90.0 4.6
RDS 5.1 5.1
PULP 0.0 0.0
WATER 94.93 96.2
100 101.4
MP % WT
SUGAR 91.8 149.9
NSDS 8.2 13.5
RDS 10.8 163
PULP 0.0 0.0
WATER 89.2 1347
100 1510

FIG. 2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SUGAR BEETS
112
HAMMER MILL
116
118
GRINDER
114
119
120
126
128
138
130
134
146
CENTRIFUGE
SCREEN 1
SCREEN 5
CENTRIFUGE
AIR
124
122
132
136
H_2O_2
148
HEATER
149
PRESS
142
140
144
FIBER
150
FILTER SCREEN
152
166
154
156
164
158
RENTENTATE UF1
pH ADJUST
- NaOH
- $Ca(OH)_2$
ULTRAFILTER 1
DIAFILTER 1
162
170
160
168
182
172
ULTRAFILTRATION 2 DIAFILTER
184
WATER
ULTRAFILTER 2
SO_2
174
180
176
178
188
186
MOLASSES
191
192
NANOFILTRATION (1 STAGE)
ELECTRODIALYSIS
ION EXCHANGE SOFTENING
190
198
193
194
PERMEATE
196
ASH/ORGANICS
SO_2
EVAPORATION
202
228
205
204
(OPTIONAL) ENZYME EXTRACTOR
206
218
CRYSTALLIZATION 1
CRYSTALLIZATION 2
208
210
220
234
216
2ND GREENS
CENTRIFUGE 1
CENTRIFUGE 2
200
212
214
222
224
WHITE SUGAR 1
WHITE SUGAR 2
MOLASSES

FIG. 3

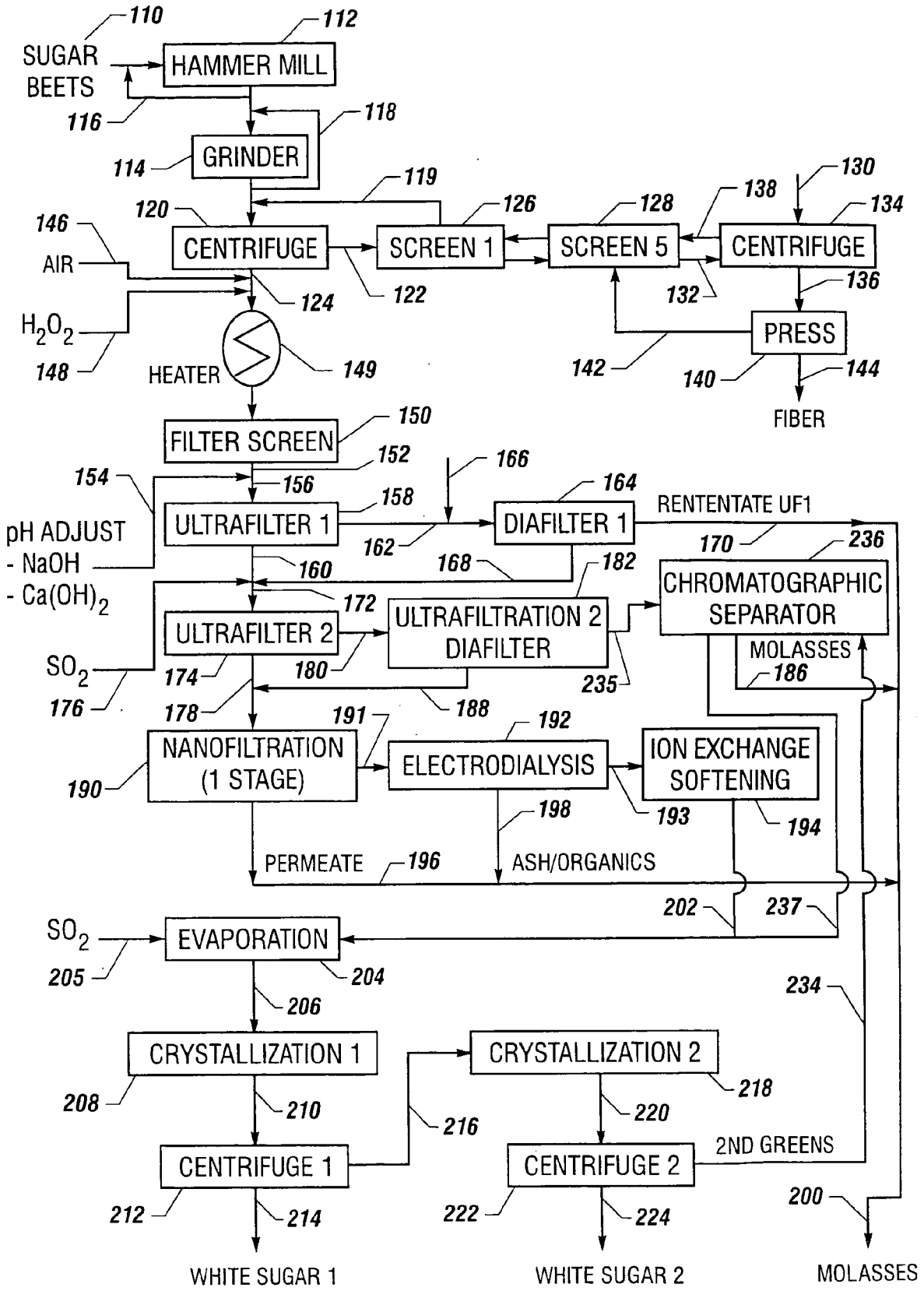

SUGAR BEETS
110
HAMMER MILL
112
116
118
GRINDER
114
119
120
CENTRIFUGE
126
SCREEN 1
128
SCREEN 5
138
130
CENTRIFUGE
134
146
AIR
124
122
132
136
H_2O_2
148
HEATER
149
PRESS
142
140
144
FIBER
FILTER SCREEN
150
152
166
154
156
164
158
ULTRAFILTER 1
DIAFILTER 1
RENTENTATE UF1
pH ADJUST
- NaOH
- $Ca(OH)_2$
162
168
182
170
236
160
CHROMATOGRAPHIC SEPARATOR
172
ULTRAFILTRATION 2 DIAFILTER
ULTRAFILTER 2
MOLASSES
186
SO_2
174
180
235
176
178
188
191
192
NANOFILTRATION (1 STAGE)
ELECTRODIALYSIS
ION EXCHANGE SOFTENING
190
198
193
194
PERMEATE
196
ASH/ORGANICS
202
237
SO_2
EVAPORATION
205
204
234
206
CRYSTALLIZATION 1
CRYSTALLIZATION 2
208
218
210
220
216
2ND GREENS
CENTRIFUGE 1
CENTRIFUGE 2
200
212
214
222
224
WHITE SUGAR 1
WHITE SUGAR 2
MOLASSES

FIG. 4

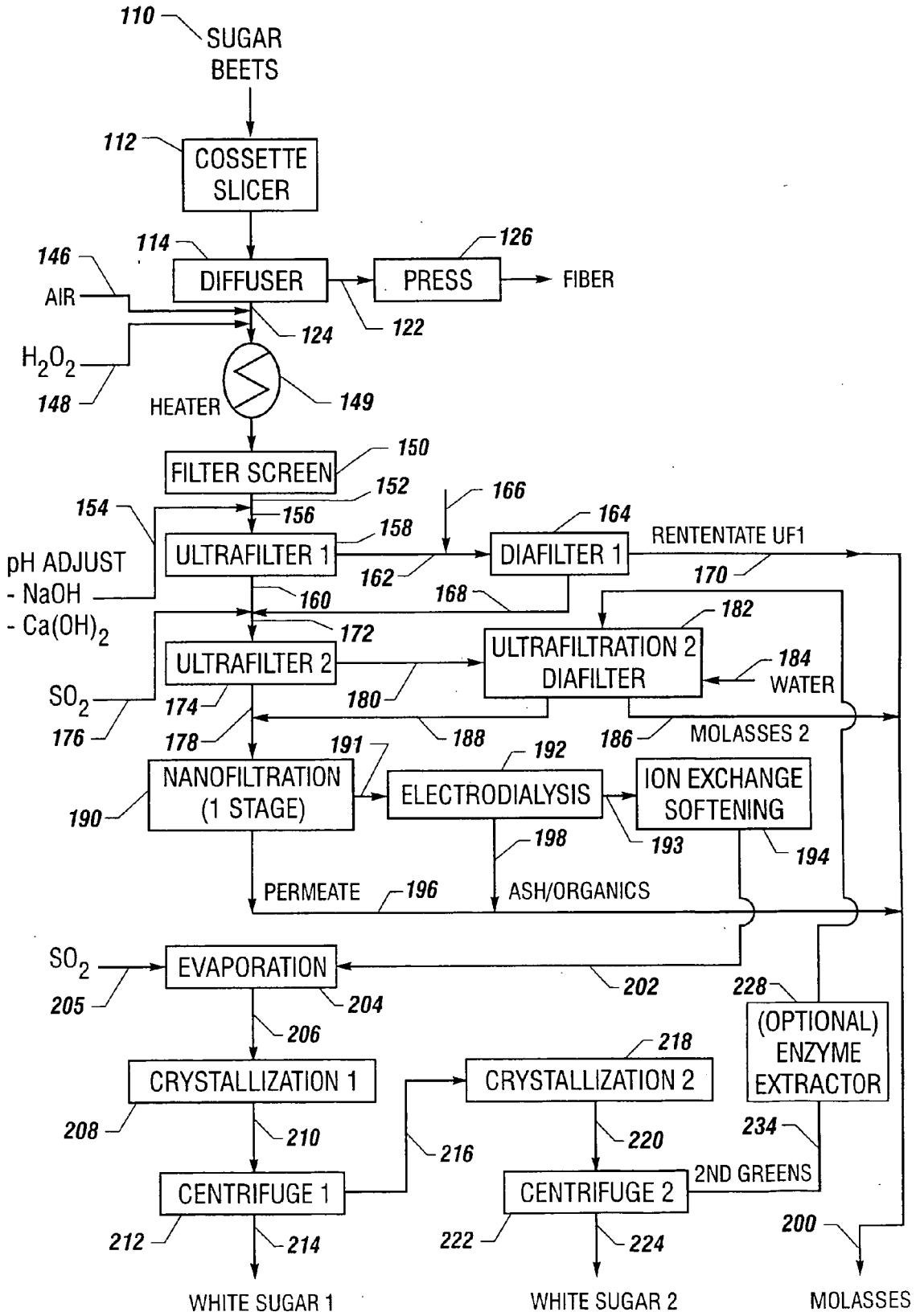
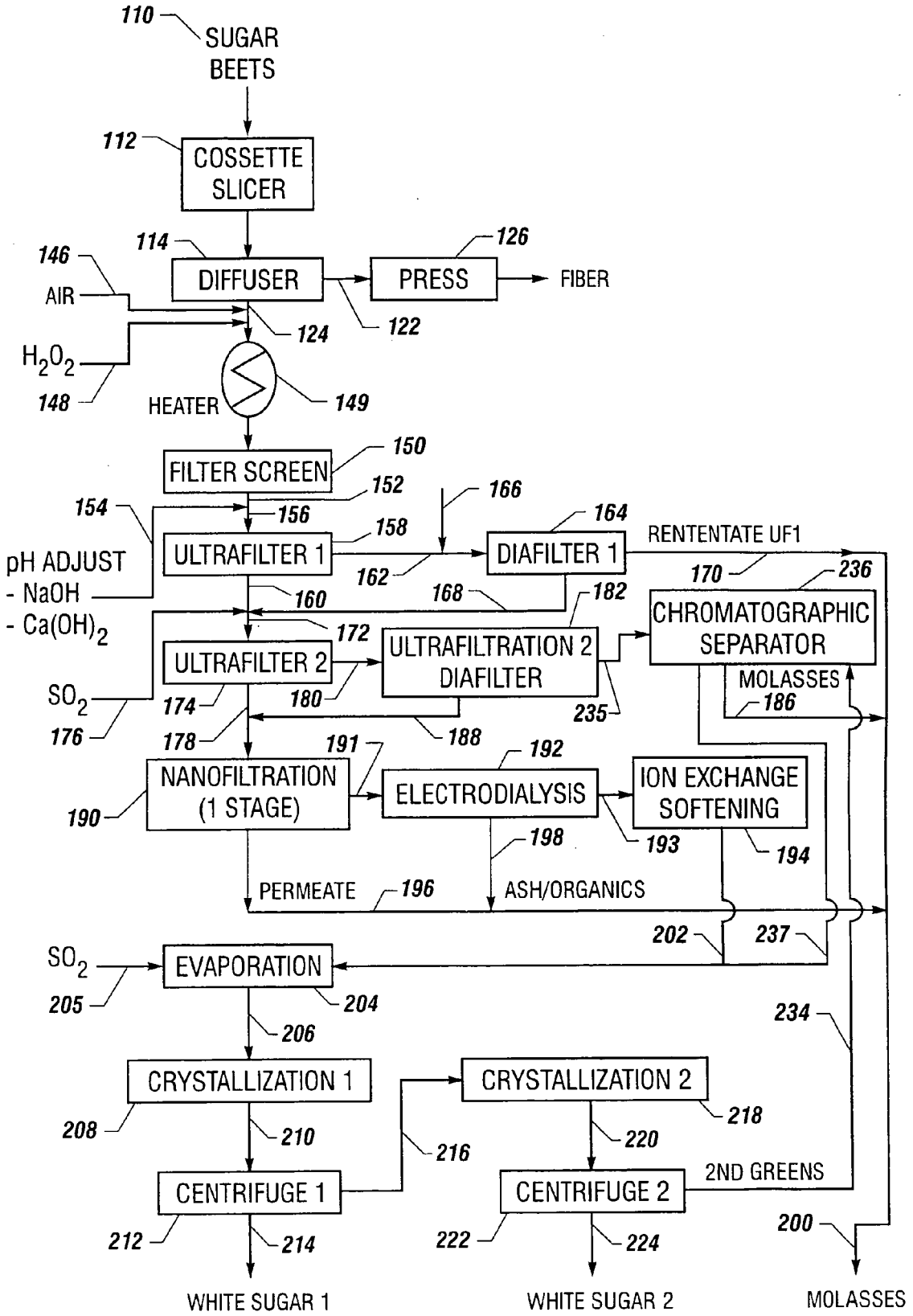

110
SUGAR BEETS
112
COSSETTE SLICER
114
DIFFUSER
126
PRESS
FIBER
146
AIR
124
122
H_2O_2
148
HEATER
149
150
FILTER SCREEN
152
154
156
166
164
158
ULTRAFILTER 1
DIAFILTER 1
RENTENTATE UF1
pH ADJUST
- NaOH
- $Ca(OH)_2$
162
168
170
160
182
172
ULTRAFILTRATION 2 DIAFILTER
184
WATER
ULTRAFILTER 2
SO_2
174
180
176
178
188
186
MOLASSES 2
191
192
NANOFILTRATION (1 STAGE)
ELECTRODIALYSIS
ION EXCHANGE SOFTENING
190
198
193
194
PERMEATE
196
ASH/ORGANICS
SO_2
EVAPORATION
202
228
205
204
206
(OPTIONAL) ENZYME EXTRACTOR
218
CRYSTALLIZATION 1
CRYSTALLIZATION 2
208
210
220
234
216
2ND GREENS
CENTRIFUGE 1
CENTRIFUGE 2
200
212
214
222
224
WHITE SUGAR 1
WHITE SUGAR 2
MOLASSES

FIG. 5


110
SUGAR BEETS
112
COSSETTE SLICER
114
DIFFUSER
126
PRESS
FIBER
146
AIR
124
122
H_2O_2
148
HEATER
149
150
FILTER SCREEN
152
154
156
166
164
RENTENTATE UF1
158
pH ADJUST
- NaOH
- $Ca(OH)_2$
ULTRAFILTER 1
162
DIAFILTER 1
170
236
160
168
182
CHROMATOGRAPHIC SEPARATOR
172
ULTRAFILTRATION 2 DIAFILTER
ULTRAFILTER 2
SO_2
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180
MOLASSES
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176
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NANOFILTRATION (1 STAGE)
ELECTRODIALYSIS
ION EXCHANGE SOFTENING
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PERMEATE
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ASH/ORGANICS
202
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SO_2
EVAPORATION
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234
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CRYSTALLIZATION 1
CRYSTALLIZATION 2
208
218
210
220
216
2ND GREENS
CENTRIFUGE 1
CENTRIFUGE 2
200
212
214
222
224
WHITE SUGAR 1
WHITE SUGAR 2
MOLASSES

FIG. 6

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

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