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PREFERENTIAL SEPARATION OF ETHANOL IN AQUEOUS SOLUTIONS

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ABSTRACT

A method of treatment of aqueous ethanol solutions, such as wine, beer or cider, by separation into at least three heterogeneous streams comprising a retentate, intermediate retentate and permeate is disclosed. Such separation conducted with the aim of preferentially increasing the concentration of ethanol in the intermediate retentate while reducing the ethanol concentration of the permeate. The intermediate retentate being a clarified, valuable product suitable for a plurality of uses that may, optionally, be recombined with the initial retentate to produce a variant of the original aqueous ethanol solution with an increased alcohol content, such as a fortified wine. The permeate being a clarified, valuable product suitable for a plurality of uses that may, optionally, be recombined with the initial retentate to produce a variant of the original aqueous ethanol solution with a reduced alcohol content, such as a low alcohol wine.

Drawings

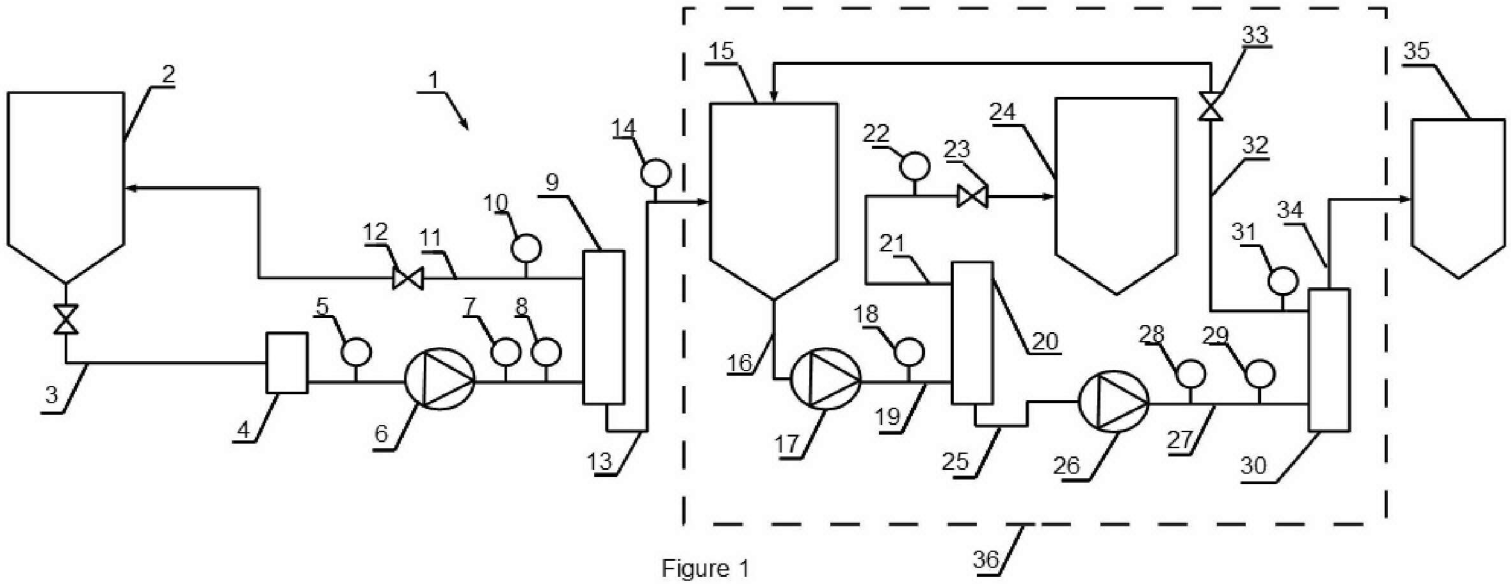

Figure 1

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PREFERENCIAL SEPERATION OF ETHANOL IN AQUEOUS SOLUTIONS

FIELD

[0001] This novel and inventive method and apparatus relates to the adjustment of the concentration of ethanol in aqueous solutions.

BACKGROUND

[0002] The fortification of wine has a history of at least 350 years and is a distinct and well established part of the beverage industry. Fortified wine products range from amongst the most expensive to the least expensive alcoholic beverages in the market.

[0003] Fortified wine type products are traditionally famed for their concentrated and intense flavours and aromas. Recently however there has been the emergence of much more subtle styles in fortified wines such as “Divas Vkat” in Australia along with a large range of flavoured wine liquors, including cream based wine liquors, which require a more neutral fortified wine base.

[0004] Traditionally, wines are fortified with grape brandy, however the additional alcohol may also be neutral spirit that has been distilled from grapes or other sources. Regional appellation laws may dictate the types of spirit that are permitted for fortification, for example, in Australia and the U.S.A. only spirit produced from grapes may be used for this purpose.

[0005] Fortifying spirit is usually purchased from external suppliers under conditions of strict regulation. In Portugal for instance, fortifying spirit for Port Wines may only be purchased from a single approved government source. The high energy costs of distillation mean that the alcohol in the spirit is usually more expensive to produce, per unit quantity, than the alcohol in the wine to which it is added. This fact together with costs of transportation, regulatory compliance and the economics of dealing with external suppliers means that the provision of fortification is an expensive and onerous part of the production of such fortified beverages.

[0006] The ethanol fuel industry has given impetus to the development of alcohol tolerant yeasts able to successfully ferment musts to alcohol concentrations above 20% ABV. However such yeasts have not been optimised for palatability and require stringent control of their growing environments which are incompatible with the techniques for producing quality wines. To date these yeast strains have not found favour in the wine industry.

[0007} Freeze concentration also known as fractional freezing, is sometimes used to increase the alcohol concentration in the production of certain beers and ciders. In the English language these products are usually known as Ice Beer or Apple Jack. The technique is very seldom used with wines. By partially freezing the beer or cider, the producer is able to remove a portion of the water from the beverage as ice, thereby increasing the concentration of the other components of the beverage including alcohol. However as the alcohol levels increase, the freezing point of the solution is lowered making the elevation of alcohol levels by any substantial amount a costly exercise.

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[0008] Reverse osmosis membrane based systems and pervaporation have been used successfully with the object of reducing the alcohol concentration in aqueous ethanol solutions to produce products such as low alcohol wine. However these systems have not been designed or optimised with the object of concentration of alcohol or the recovery of the alcohol that has been removed.

[0009] There are therefore a number of compelling commercial and regulatory advantages, offering considerable benefits, to a manufacturer able to selectively separate and concentrate alcohol from an aqueous solutions in such a manner that it:

1. constitutes a valuable product in its own right;
2. produces a reduced alcohol permeate that constitutes a valuable product in its own right;
3. may be used to enhance the concentration of ethanol in the original retentate to produce fortified type products without recourse to the use of fortifying spirit;
4. may be removed permitting the recombination of the original retentate and final permeate to produce an aqueous ethanol solution with a reduced alcohol level such as a low alcohol wine.

SUMMARY

[0010] The object of the present invention is to provide a novel method and apparatus for the increase or decrease of the concentration of ethanol in aqueous solutions such as wine, beer or cider by separating and differentially removing portions of the water or alcohol present in the solution via a reverse osmosis membrane process having a number of concentrating steps.

[0011] According to the present invention, there is provided a method of separation of aqueous ethanol solutions into heterogeneous streams and treatment thereof to increase the concentration of alcohol in at least one predetermined stream thereof including the steps of:

[0012] processing the aqueous ethanol solution by reverse osmosis or nanofiltration for producing a retentate and a raw permeate. The said raw permeate containing largely water and ethanol. The said retentate containing any flavouring, colouring and other compounds of higher molecular weight present in the initial aqueous ethanol solution;

[0013] processing the raw permeate by reverse osmosis with one or more following membranes sets. Such membrane sets having the characteristic of high ethanol exclusion, thereby producing an intermediate retentate, or retentates, with enhanced ethanol concentration levels and a permeate or permeates with depleted ethanol concentration levels; and

[0014] optionally combining the retentate from the first set of membranes with an intermediate retentate produced by the following membrane or membranes. Thereby producing a treated aqueous ethanol solution in which the ethanol level is enhanced, such as a fortified wine. Or, optionally combining the retentate from the first set of membranes with the final permeate produced by the following membrane or membranes. Thereby producing a treated aqueous ethanol solution in which the ethanol level is reduced, such as a low alcohol wine.

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[0015] The invention also provides apparatus for such adjustment of the concentration of ethanol in aqueous solutions via a reverse osmosis membrane process having a number of concentrating steps. The apparatus including:

[0016] a first processing stage having a reverse osmosis unit or nanofiltration unit having a retentate outlet and a permeate outlet;

[0017] a pump for supplying the aqueous ethanol solution to be treated under pressure to said first processing stage. Whereby retentate is produced at said retentate outlet and raw permeate containing largely ethanol and water is produced at the permeate outlet;

[0018] a second processing stage which includes at least one reverse osmosis process, the second processing stage having an inlet for receiving said raw permeate, a pump to pressurise said raw permeate and one or more reverse osmosis membranes. Said membranes being operable to remove at least a portion of the ethanol from the said raw permeate and produce an intermediate retentate at an outlet with increased ethanol concentration and a permeate at an outlet of the second processing stage having a reduced ethanol concentration; and optionally

[0019] means for combining said retentate from the first processing stage and said concentrated ethanol solution from the second processing stage or the said depleted ethanol solution from the second processing stage to thereby produce treated aqueous ethanol solution in which the alcohol concentration is increased or decreased.

BRIEF DESCRIPTION OF THE DRAWINGS

[0020] The invention will now be further described with reference to the accompanying drawings, in which:

[0021] FIG. 1 is a schematic diagram of a preferred embodiment of the invention.

DESCRIPTION OF EMBODIMENTS

[0022] Referring first to FIG. 1, there is shown an apparatus 1 for the concentration of aqueous ethanol solutions having an initial ethanol concentration of between 4% and 16% Alcohol By Volume (ABV). The apparatus includes a storage tank 2 for the aqueous ethanol solution to be treated. An outlet line 3 passes to a screen filter 4 which has a 1 mm screen for removal of larger solid components from the aqueous ethanol solution. The aqueous ethanol solution is then pumped to a first processing stage 9 by means of a pump 6. A flow meter 5 is connected between the filter 4 and the pump 6 and pressure and temperature sensors 7 and 8 are connected between the pump 6 and the first processing stage 9, as indicated.

[0023] The first processing stage 9 may be a reverse osmosis unit or a bank of filters having nanofiltration membranes therein. As will be apparent to those skilled in the art, with modern membranes there is little technical difference between nanofiltration and reverse osmosis. The main difference is the relative permeability of the membranes to components of varying molecular weight and charge.

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[0024] The first processing stage 9 is arranged to produce a permeate which includes compounds below a predetermined molecular weight such as 150 to 300. The permeate stream which consists of water, ethanol, certain low molecular weight organic compounds and some monovalent ions, is generated. It does not contain colour and has little flavour. To be effective the membranes need to pass water and ethanol but reject most other solution components.

[0025] Membranes used in the first processing stage may be of the reverse osmosis or nanofiltration type.

[0026] One such membrane is GE Osmonics Vinopro 8040C-30D. This is a proprietary thin film nanofiltration membrane in a spiral wound construction. It is available in various size configurations which may be chosen according to performance requirements. The advantage of the spiral wound arrangement is that large filter surface areas may be available in a relatively small overall volume.

[0027] Another such membrane is GE Osmonics Vinocon-1 8040C-30D. This is a proprietary thin film reverse osmosis membrane in a spiral wound construction. It is available in various size configurations which may be chosen according to performance requirements.

[0028] Membrane performance is typically measured in flux of permeate and relative passage and rejection of the compounds to be separated. It varies with the osmotic pressure, clarity and temperature of the aqueous ethanol solution being treated.

[0029] Preferably the temperature of the aqueous ethanol solution should be between 15° C. and 25° C. to promote flux without compromising any thermally sensitive characteristics of the product being processed.

[0030] Preferably, the operating pressure should be between 1,000 and 7,500 kPa. Higher pressures may be injurious to the membranes. At lower pressures, the flux is so low that large membrane surface areas are required for satisfactory system performance.

[0031] The membrane performance can to some degree be regulated by pressure with regard to its selectivity i.e. rejection of higher molecular weight compounds may be improved by operation at higher pressure.

[0032] Preferably the aqueous ethanol solution in the tank 2 should be initially clarified using known techniques such that grape or other suspended solids do not foul the membranes. For optimum membrane performance, the clarity as measured by turbidity should be less than 50 NTU.

[0033] Under these conditions, the permeate flux would be 15-20 litres per square metre of membrane area per hour.

[0034] A typical operating configuration of four membranes comprises approximately 120 square metres of membrane surface area and permeate flux is 1,800 to 2,400 litres per hour.

[0035] The first processing stage 9 includes a retentate outlet line 11 which is arranged to return the retentate back to the aqueous ethanol solution in the tank 2, as shown. The line 11 includes a back pressure control valve 12 and pressure sensor 10 which are arranged to facilitate operational control of the pressure drop across the first stage 9.

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[0036] The permeate passes to a buffer tank 15 via a permeate line 13. A second flow meter 14 is connected in the line 13 so as to sense the flow rate in the line 13.

[0037] Generally speaking, the second processing step 36 is arranged to increase the concentration of ethanol in the retentate of the two processing stages 20 and 30.

[0038] The processing step 36 consists of two reverse osmosis stages 20 and 30 and is arranged to produce a permeate which as far as possible includes only compounds below a predetermined molecular weight such as 35 to 45 thereby retaining ethanol in the retentate. Reverse osmosis membranes enabling over 80% ethanol retention do currently exist, but the maximum concentration extent is limited to about 25% ABV. Beyond this concentration the retention rate decreases rapidly. Technical improvements and innovation in membrane technology can be expected to improve both ethanol retention and maximum concentration performance in the future.

[0039] The aqueous ethanol solution is pumped from the buffer tank 15 via an outlet line 16 to a processing stage 20 by means of a pump 17. A pressure sensor 18 is connected in the line 19 between the pump 17 and the processing stage 20, as indicated.

[0040] The processing stage 20 includes a retentate outlet line 21 which is arranged to convey the retentate to the tank 24, as shown. The line 21 includes a back pressure control valve 23 and pressure sensor 22 which are arranged to facilitate operational control of the pressure drop across the processing stage 20.

[0041] For an aqueous ethanol solution with an initial alcohol concentration of 14% ABV the processing stage 20 produces a retentate with an alcohol concentration of about 22% ABV. Because the ethanol retention of currently available reverse osmosis membranes is limited, the permeate produced from the processing stage 20 will contain about 10.5% ABV.

[0042] The permeate from the processing stage 20 is conveyed via an outlet line 25 to a buffer tank, not shown, and pump 26 to a following processing stage 30 in order to recover further alcohol from it.

[0043] The processing stage 30 includes a retentate outlet line 32 which is arranged to convey the retentate to the buffer tank 15, for reprocessing as shown. The line 32 includes a back pressure control valve 33 and pressure sensor 31 which are arranged to facilitate operational control of the pressure drop across the processing stage 30.

[0044] The processing stage 30 produces a retentate with an alcohol concentration of about 13% ABV. Because the ethanol retention of currently available reverse osmosis membranes is limited, the permeate produced from the processing stage 30 will contain about 2.1% ABV.

[0045] The permeate produced from the processing stage 30 is conveyed via an outlet line 34 to a collection tank 35. The permeate collected in the tank 35 may be put to a variety of uses, including the further extraction of ethanol using a variation on the processes described in the present document, dependent upon the economic viability of such further extraction.

[0046] The most suitable form will be determined by the particular requirements of the application such as the need for equipment mobility, the availability of holding vessels and the relative values, and intended use, of the output streams.

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[0047] Subsequent analysis of the permeate and retentate before and after each treatment stage would allow verification and calibration of the process.

[0048] Alternatively, measurements could be by means of alcoholic strength detectors, such as a density meter, and flow or level meters which would monitor the alcoholic strength and volume of the various streams. Output from these devices could be processed by programmable logic controller (not shown), and operating parameters such as pressures and flow rates could be optimised.

[0049] The process can be continued until a predetermined amount of the ethanol has been concentrated in the solution contained in tank 24. This can be monitored by observation of the level in the tank 24 and measurement of the alcoholic concentration of the solution in the tank 24.

[0050] If the object of the treatment is the fortification of the initial aqueous ethanol solution contained in the tank 2 then the contents of the tank 24, having reached the predetermined volume and concentration in relation to the contents of tank 2 to achieve the required fortification, may be recombined with the contents of the tank 2 to complete the process.

[0051] If the object of the treatment is to reduce the alcohol concentration of the initial aqueous ethanol solution contained in the tank 2 then the contents of the tank 35, having reached the predetermined volume and concentration in relation to the contents of tank 2 to achieve the required reduction, may be recombined with the contents of the tank 2 to complete the process.

[0052] It will be apparent to those skilled in the art that various configurations of energy recovery systems, including pressure exchangers, may usefully be deployed in the apparatus 1 to increase the energy efficiency thereof.

[0053] Many other modifications will be apparent to those skilled in the art without departing from the spirit and scope of the invention.

Editorial Note

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Claims.

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I claim:

1. A method of treatment of aqueous ethanol solutions, such as wine, to reduce or increase the concentration of the alcohol component thereof including the steps of:

i. processing the aqueous ethanol solution by reverse osmosis or nanofiltration for producing an initial retentate, an intermediate retentate, and a permeate, the intermediate retentate having increased alcohol level an the permeate containing a reduced alcohol content;

ii. combining the initial retentate with the permeate for producing treated product in which the alcohol component is reduced;

iii. combining the initial retentate with the intermediate retentate for producing treated product in which the alcohol component is increased.

2. A method as claimed in claim 1 including the steps of determining the alcohol level of the intermediate retentate and permeate in order to monitor the levels of said alcohol component.
3. A method as claimed in claim 1 including the step of storing the aqueous ethanol solution to be treated in a tank and the method includes the step of returning the combined retentate and permeate to said tank.
4. A method as claimed in claim 1 including the step of storing the aqueous ethanol solution to be treated in a tank and the method includes the step of returning the combined retentate and intermediate retentate to said tank.
5. A method as claimed in claim 1 including the step of storing the aqueous ethanol solution to be treated in a tank and the method includes the step of storing the intermediate retentate in a second tank and the permiate in a third tank.
6. A method as claimed in claim 7 wherein the aqueous ethanol solution from the tanks is reprocessed.
7. A method as claimed in claim 1 wherein the aqueous ethanol solution is clarified prior to step (i).
8. A method as claimed in claim 9 wherein the turbidity of the aqueous ethanol solution is less than 50 NTU after clarification.
9. A method as claimed in claim 1 wherein the step of processing the aqueous ethanol solution by reverse osmosis or nano filtration includes the step of passing the wine under pressure to a filtration unit which includes one or more membranes.
10. A method as claimed in claim 11 wherein the membrane or membranes are selected to pass said predetermined component to said permeate.
11. A method as claimed in claim 11 wherein the membrane or membranes pass compounds having a predetermined nominal molecular weight.
12. A method as claimed in claim 13 wherein said nominal molecular weight is in the range 40 to 300.
13. Wine treated by the method of claim 1.
14. Apparatus for treatment of an aqueous ethanol solutions, such as wine, to reduce or increase the concentration of the alcohol component thereof including: a first processing stage having a reverse osmosis unit or nanofiltration unit having a retentate outlet and a permeate outlet; a pump for supplying aqueous ethanol solution to be treated under pressure to said first processing stage whereby retentate is produced at said retentate outlet and a permeate, which becomes the intermediate retentate, at the permeate outlet; a second processing stage having a reverse osmosis unit unit having a retentate outlet and a permeate outlet; a pump for supplying aqueous ethanol solution to be treated under pressure to said second

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processing stage whereby retentate, which becomes the intermediate retentate, is produced at said retentate outlet and permeate is produced at the permeate outlet.

15. Apparatus as claimed in claim 17 including means for monitoring the alcohol levels in the apparatus.
16. Apparatus as claimed in claim 17 including a storage tank for storing aqueous ethanol solution to be treated and wherein said means for combining said retentate and said final permeate is operable to return the combined retentate and final permeate to said tank.

17. Apparatus as claimed in claim 17 including a storage tank for storing aqueous ethanol solution to be treated and wherein said means for combining said retentate and said intermediate retentate is operable to return the combined retentate and intermediate retentate to said tank.
18. Apparatus as claimed in claim 17 wherein the reverse osmosis unit or the nanofiltration units includes membranes which are operable to pass compounds having a predetermined nominal molecular weight.
19. Apparatus as claimed in claim 22 wherein said nominal molecular weight is in the range from 40 to 300.
20. Apparatus as claimed in claim 22 wherein the first membrane set is of type 4040C-30D VinoPro and the second membrane set is of type GE P/N 3056651 Model – AD-90.

Drawings

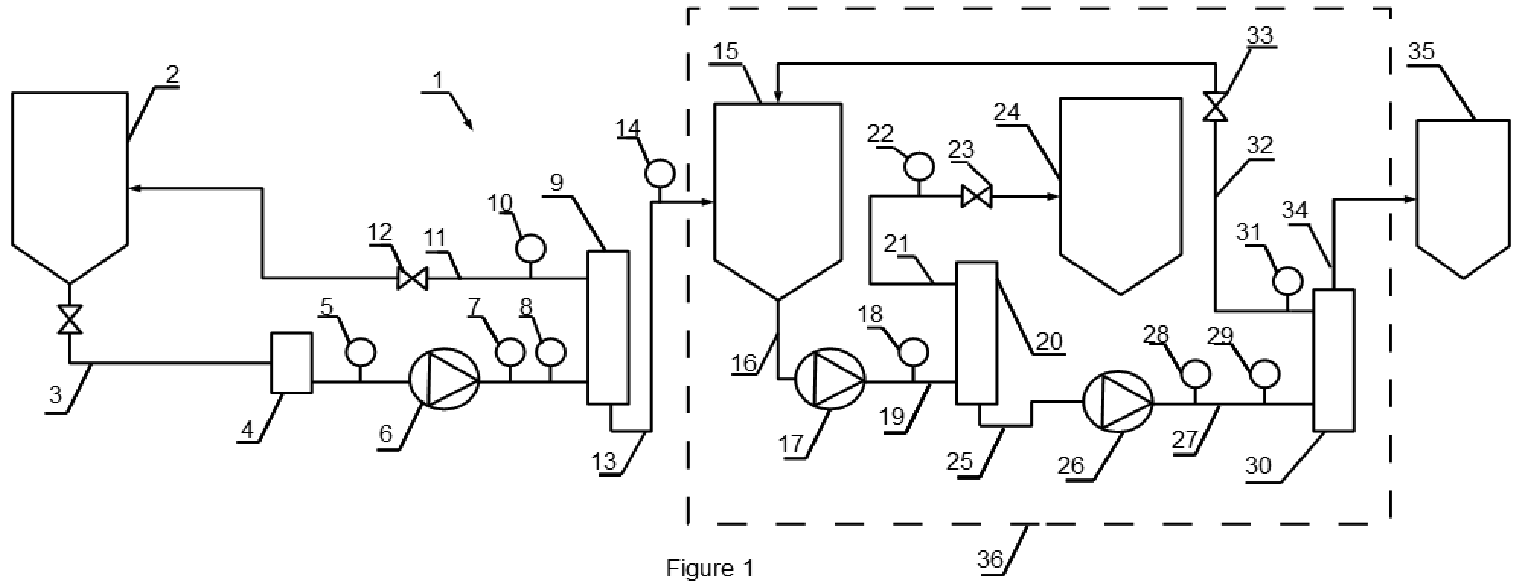

Figure 1