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- (73) Patenthaver: **Praj Industries Limited, Praj Tower , 274-275 Hinjewadi Road , Bhumkar Chowk, Pune, Hinjewadi 411057, Indien**
- (72) Opfinder: **DESHPANDE, Ghansham Baburao, Praj Industries Limited, Praj House Bavdhan, Pune 411021, Maharashtra, Indien**
RATHI, Shrikant Subhash, Praj Industries Limited, Praj House Bavdhan, Pune 411021, Maharashtra, Indien
DESHPANDE, Devdatta Krishna, Praj Industries Limited, Praj House Bavdhan, Pune 411021, Maharashtra, Indien
PIMPULKAR, Vijaykumar Surendranath, Praj Industries Limited, Praj House Bavdhan, Pune 411021, Maharashtra, Indien
BHOSALE, Abhijit Anil, Praj Industries Limited, Praj House Bavdhan, Pune 411021, Maharashtra, Indien
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DESCRIPTION

FIELD OF THE INVENTION

[0001] The invention relates to a process and apparatus for the preparation of ethanol products and more particularly to low steam requiring distillation of ethanol from a feedstock containing ethanol of at least 3% by volume using a series of specialized columns and efficiently heat integrated processes. Further the invention relates to preparation of different grades of ethanol products having minimum levels of congeners suitable for potable use.

BACKGROUND

[0002] Many alcoholic beverages are produced comprising one or more of the steps of [1] producing ethanol by fermentation of a carbohydrate-rich feedstock to produce a fermented wash having concentration of about 2 to 23% alcohol by volume [ABV]; [2] distilling the product of fermentation at elevated temperatures to produce ethanol products like rectified spirit, neutral spirit or absolute alcohol and; [3] aging the ethanol spirits until it possesses desired flavour, aroma, and colour characteristics. The commercial production of alcohol by distillation has been in widespread operation for many centuries.

[0003] The distillation is a known technique for purification of a liquid substance and involves vaporizing the substance at its boiling point, condensing the vapour and collecting the purified form as a condensate. Distillation is useful for separating a mixture when the components have different boiling points. Several kinds of distillation techniques for binary or multi-component mixtures are described and practiced in the art, for example: (1) simple, (2) vacuum or reduced pressure, (3) fractional and (4) steam distillation.

[0004] To produce rectified spirit from a fermented wash, the wash is distilled in the one or multiple distillation columns to produce a product with ethanol content of about 93% ABV. This spirit contains various congeners like fusel oils, higher alcohols, acids, etc which are carried over during the distillation process.

[0005] On the other hand, a double distillation process is carried out for manufacturing neutral spirit in which a fermented wash is distilled to about 95% ABV and is then subjected to a process of hydro-selection or hydro-extraction in which ethanol is again diluted with water between about three to about twelve times and redistilled to the azeotropic concentration of ethanol. This process helps in removing the congeners that are unwanted in neutral spirit.

[0006] The complete removal of water from ethanol gives anhydrous alcohol, which is widely used in industry as a solvent in the synthesis of paints, pharmaceutical intermediaries, cosmetics, perfumes, and other products. Anhydrous ethanol is also an important component

in alternative fuels such as gasohol or it can also be used as an oxygenate in gasoline. To make anhydrous ethanol, the azeotrope of ethanol-water is broken to achieve the desired strength of more than 99% ABV by using one of the dehydration methods like: 1] molecular sieve dehydration, 2] azeotropic distillation, or 3] membrane dehydration. This makes traditional distillation to obtain anhydrous ethanol a costly process requiring high amounts of energy to obtain pure ethanol. Other processes that can be used to obtain anhydrous ethanol include extractive distillation and salt rectification. However, these processes require high energy input resulting in expensive anhydrous ethanol products. Currently, typically about 3-4 kilograms of steam is used to prepare about one litre of high quality ethanol product, leading to need of new processes and apparatuses more energy efficient to achieve less steam consumption and more economic to operate.

[0007] Several methods have been described in the art, for example, the patent US4784868 describes a method of producing a potable spirit which comprises obtaining ethanol substantially free of congeners and water and then fractionally distilling this mixture to produce substantially water-free potable ethanol. However, this process uses liquid CO₂ in dehydration process and is not practical and economic in many situations. Therefore, there is further need for effective and economic processes for the preparation of high quality ethanol products, especially absolute ethanol for a variety of applications from potable use to vehicular fuels.

[0008] US 5 035 776 A discloses a thermally-integrated extractive distillation process for recovering anhydrous ethanol from fermentation or synthetic feedstocks that has a distillation train of four columns. Two columns are preconcentrators operated in parallel. The remaining columns are an extractive distillation dehydrating tower, and an entrainer-recovery column. The two preconcentrators and the dehydrating tower are operated at three successively increasing pressures so that the condensing vapors of the overhead product of the dehydrating tower supply the necessary heat to the reboiler of the intermediate-pressure preconcentrator. The overhead vapors of this preconcentrator are, in turn, used to supply the required heat to the reboiler of the lowest-pressure preconcentrator. The bottom product from each preconcentrator is used to preheat the dilute feed. Additional energy savings are accomplished by the appropriate heat exchange between the various feeds, overheads, and bottoms.

BRIEF DESCRIPTION

[0009] The present invention provides a process for separating ethanol from a fermented wash containing minimum of 2% ABV to various high quality ethanol products like anhydrous ethanol to extra neutral ethanol having minimum qualities of congeners with no remarkable odour or taste.

[0010] In one embodiment of the present invention, the process of separation of ethanol from fermented wash may be divided into seven stages: 1] primary distillation, 2] aldehyde distillation, 3] low boiler distillation, 4] rectification distillation, 5] dehydration, 6] high boiler

distillation and 7] simmering distillation.

[0011] According to the present invention, a distillation method for separating ethanol from a dilute ethanol containing feedstock comprising: dividing the feedstock into two feed streams of unequal size; preheating both the larger and the smaller of said feed streams; introducing said preheated feed streams in parallel into a pair of analyzer columns; maintaining a higher pressure in first analyzer column receiving said larger feed stream than in second analyzer column receiving said smaller feed stream; withdrawing a first hydrous ethanol stream from the upper portion of said analyzer columns and feeding it to an aldehyde column to separate an aldehyde free ethanol stream at the bottom portion and a technical alcohol stream at the upper portion of the column; mixing about three to twelve parts of water to one part of said aldehyde free ethanol stream, preheating it and subjecting it to a low boiler column to separate a second hydrous ethanol stream from the lower portion and a technical alcohol stream from the upper portion of the column; preheating said second hydrous ethanol stream and subjecting it to a rectifier column to obtain a rectified ethanol stream; dehydrating said rectified ethanol stream using a dehydration unit to obtain an anhydrous ethanol stream; introducing said anhydrous ethanol stream to a high boiler column to separate a technical alcohol stream at the bottom portion and a final anhydrous ethanol stream at the upper portion of the column; and introducing said final anhydrous ethanol stream to a simmering column to obtain ethanol product streams by optionally adding water to said final anhydrous ethanol stream in various amounts.

[0012] According to the present invention, a distillation apparatus for preparation/ recovering ethanol from a dilute ethanol containing feedstock comprising, in combination: a provision for splitting the feedstock into two feed streams of unequal size; a provision for preheating both the larger and the smaller of said feed streams; a high pressure first analyzer column and a low pressure second analyzer column; a provision for introducing the larger of said feed streams into said first analyzer column and a provision for introducing the smaller of said feed streams into said second analyzer column; a first reboiler means for condensing alcohol vapours from dehydration unit to supply the heat required in said first analyzer column; a second reboiler means for condensing overhead vapours from a rectifier column downstream to supply the heat required in said first analyzer column; a third reboiler means for condensing overhead vapours from said first analyzer column to supply the heat required in said second analyzer column; a provision for withdrawing of first hydrous ethanol streams from an upper portion of said analyzer columns; a provision for preheating and introducing said hydrous ethanol streams into an aldehyde column; a provision for withdrawing of an aldehyde free ethanol stream from a bottom portion of said aldehyde column; a provision for preheating and introducing said aldehyde free ethanol stream into said rectifier column; a provision for withdrawing of a rectified ethanol stream from an upper portion of said rectifier column; a provision for introducing an elemental hydroxide to said rectified ethanol stream to adjust its pH; a provision for introducing said rectified ethanol stream into a dehydration unit; a provision for withdrawing of an anhydrous ethanol stream from said dehydration unit; a provision for introducing an inorganic oxidising agent to said anhydrous ethanol stream; means for introducing said anhydrous ethanol stream into a simmering column having a provision to

optionally mix water; and a provision for withdrawing of ethanol product streams from said simmering column.

BRIEF DESCRIPTION OF THE DRAWINGS

[0013] Particular examples of methods in accordance with this invention will now be described with reference to accompanying drawings, in which:

FIGURE 1 is a schematic diagram of the mass flow in the disclosed process. A fermented wash is split and fed to said analyzer columns. The distillate is then fed in a series to aldehyde column, low boiler column and rectification column. Then rectified stream is dehydrated in a dehydration unit and then subjected to high boiler column and at the end to a simmering column to finally get neutral spirit of desired quality. Different types of feints are obtained from different columns forming technical alcohol products of different grades.

FIGURE 2 is a block diagram of the energy flow in the disclosed invention. The first analyzer column gets energy in the form of unused [excess] energy from rectification column and dehydration unit. The second analyzer column gets energy in the form of unused energy from said first analyzer column. The aldehyde analyzer column gets energy in the form of unused energy from both the said analyzer columns. The unused energy of said low boiler column is supplied to said high boiler column. In addition, fresh energy in the form of steam is supplied to said low boiler column, said rectification column and said dehydration unit at need.

FIGURE 3 is an exemplary plan of the invention showing several features that control the process of distillation of ethanol from a fermented wash. It also depicts the efficient energy integration achieved through reuse of the heat energy left unused in the downstream steps to run the unit operations at upstream steps. The whole system may be divided into seven units, namely: 1] primary distillation unit, 2] aldehyde distillation unit, 3] low boiler distillation unit, 4] rectification distillation unit, 5] dehydration unit, 6] high boiler distillation unit, and 7] simmering unit. The unused energy left in the form of vapours from said rectification distillation unit is integrated to run said first analyzer column in said primary distillation unit. Further the unused energy left in the form of vapours from said dehydration unit is integrated with said first analyzer column. The unused energy left in the form of vapours from said low boiler distillation unit is integrated with said high boiler distillation unit. The unused energy left in the form of vapours from said primary distillation unit is integrated with said aldehyde column.

DETAILED DESCRIPTION

[0014] In one embodiment of the present invention, a fermented wash with ethanol concentration of between 2 to 23% ABV, preferably 6 to 14% ABV is obtained from yeast fermentation of a carbohydrate rich feedstock like grains, cereals, tubers, molasses, sugarcane

juice, lignocellulosic materials or synthetic ethanol. This wash is then split into two feed streams of unequal size, a larger feed stream comprising about 55 to 70% of the total wash and a smaller feed stream comprising remaining about 30 to 45% of the wash. These two feed streams have a temperature of about 30 °C. Before subjecting it to the primary distillation unit these feed streams are preheated to about 76 °C by plate-type heat exchangers. The said heat exchangers receive high energy spent wash of corresponding analyzer columns as heat donating liquid to preheat said fermented wash before subjecting it to the process of primary distillation. The said primary distillation unit comprises two analyzer columns operating at different working pressures. The first analyzer column receiving said larger feed stream operates at a higher pressure and temperature than the second analyzer column receiving said smaller feed stream. In this type of split primary distillation process said first analyzer column typically provide unused energy in the form of vapours to said second analyzer column, which is operated at relatively lower pressure and temperature than said first analyzer column. This split distillation using minimum amount of energy to separate ethanol from said fermentation wash forms an element of novelty in view of the total energy consumption per unit of final ethanol product produced. The ethanol containing streams obtained from the upper portions of said analyzer columns are combined to form first hydrous ethanol streams. The spent wash obtained from said analyzer columns possess excess unused energy, which is used in said heat exchangers to preheat said feed stream as described herein. The said analyzer columns of the invention are also equipped with degassing columns at the top, which are heat integrated by the vapours of said analyzer columns. It is understood in the art the utility of degassing column and hence not specifically depicted herein. These degassing columns remove dissolved gases like CO₂ present in fermented wash before its entry into said analyzer columns.

[0015] In another embodiment of the present invention, the process of preparation of the high quality ethanol as disclosed herein comprises of seven steps, namely: 1] primary distillation unit, 2] aldehyde distillation unit, 3] low boiler distillation unit, 4] rectification distillation unit, 5] dehydration unit, 6] high boiler distillation unit, and 7] simmering unit. The said primary distillation unit is described herein above is also called split distillation and it forms the first important step of the disclosed invention. In second step, a part of said first hydrous ethanol stream is subjected to an aldehyde distillation leading to the formation of an aldehyde free ethanol stream. The said aldehyde distillation process removes one or more aldehyde components of the stream making it free of any aldehyde taste or odour. Herein high feints obtained form a grade of technical alcohol used for a variety of non-food purposes. In third step, said aldehyde free ethanol stream along with the vapour condensate from both the analyzer columns is subjected to a low boiler distillation leading to the formation of a second hydrous ethanol stream. Before subjecting said aldehyde free ethanol stream to said low boiler distillation, it is diluted with about three to twelve parts of water or spent lees obtained downstream. Herein said low boiler distillation process removes one or more low boiling components than ethanol of the stream making it free of any low boilers like ethyl acetate and acetaldehyde and its associated taste or odour. Herein high feints obtained forms another grade of technical alcohol used for a variety of non-food purposes. In fourth step, said second hydrous ethanol stream is subjected to a rectification distillation leading to the formation of a

rectified ethanol stream. Herein said rectified ethanol stream is treated with an elemental hydroxide like sodium hydroxide, potassium hydroxide, calcium hydroxide or equivalent other hydroxide to improve the pH of said stream to between pH 7 to pH 11. This treatment substantially improves the quality of said stream eradicating any acidity associated with it. The said rectification distillation process improves the ethanol content of the stream to about 93% ABV. This step further removes the congeners like acids and fusel oils present in the stream leading to a high quality of ethanol in said rectified ethanol stream. Herein high feints obtained forms another grade of technical alcohol, while the spent lees obtained are used as diluents in the third step described herein above. In fifth step, said rectified ethanol stream is subjected to a dehydration process leading to the formation of an anhydrous ethanol stream. This dehydration process improves the ethanol content of the stream to about 99% ABV. This dehydration is achieved by means of one or more of methods like membrane separation, molecular sieve dehydration, extractive distillation or adsorption separation of water or combination thereof. In sixth step, said anhydrous ethanol stream is subjected to a high boiler distillation leading to the formation of a final anhydrous ethanol stream. Herein said final anhydrous ethanol stream is treated with an inorganic oxidising agent like potassium permanganate, hydrogen peroxide, sodium percarbonate or equivalent chemical to remove other alcoholic impurities of higher primary and secondary alcohols like butanol, propanol, pentanol, ketones and other chemicals that adversely affect the organoleptic properties of the final products, leading to substantially improvement in the final quality of said stream. Herein further said high boiler distillation process removes one or more high boiling components than ethanol of the stream making it free of any high boilers like butanol and its associated taste or odour. Herein low feints obtained forms another grade of technical alcohol. In seventh step, said final anhydrous ethanol stream is subjected to a simmering distillation leading to the formation of a final ethanol product. Herein said simmering distillation process removes methanol and one or more sulphide components from the stream making it free of any associated taste or odour. The said simmering column has copper internal components, while other process equipments are made of stainless steel or other suitable materials. Herein high feints obtained forms another grade of technical alcohol. In the simmering process of the disclosed invention, various amount of water of dilution may be added before or after the production process to achieve different types of ethanol products based on the final content of ethanol. Another novelty of the process is that the final iso-propanol amount is reduced at least to 40% of the initial amount in the feed streams.

[0016] In yet another embodiment of the invention, in said primary split distillation unit, said second analyzer column operating at lower pressure operates at a pressure between about 0.1 to about 1 bar[a], preferably about 0.2 bar[a]. Any unused energy left in said first analyzer column is used to drive said second analyzer column and said aldehyde column. In said rectification distillation unit, said rectification column operates at a pressure between about 1 to about 10 bar[a], preferably between about 2 to about 3 bar[a]. Herein the column pressure is so adjusted that it acts as the heat source for the first analyzer column operating upstream in the process.

[0017] In further embodiment of the disclosed invention as illustrated in FIGURE 3 the steps of

said process are: 1] the fermented wash (1) is divided unequally into two feed streams (2 and 3), each feed stream is then preheated to desired temperature by heat exchangers (39) and enters into said first analyzer column (31) and said second analyzer column (30); 2] then a part of first hydrous ethanol stream (8) from said second analyzer column (30) is directed to said aldehyde column (32), while remaining part (6) is condensed (40) and directed (6) to the dilution tank (42), similarly first hydrous ethanol stream (9) from said first analyzer column (30) is directed to the column (32); 3] said aldehyde free ethanol stream (10) is directed the dilution tank (42), in the dilution tank the stream is dilution to three to twelve times with water or spent lees (15) and this stream (16) is directed to said low boiler column (33); 4] said second hydrous ethanol stream (17) obtained from said low boiler column (33) is preheated in a heat exchanger (39) by the energy of spent lees (15) of the column (34) and is directed to said rectification column (34) to obtain said rectified ethanol stream (28); 5] said rectified ethanol stream (28) is then directed to said dehydration unit (35) to obtain said anhydrous ethanol stream (18); 6] said anhydrous ethanol stream (18) is then directed to said high boiler column (36) to obtain said final anhydrous ethanol stream (22); and 7] said final anhydrous ethanol stream (22) is then directed to said simmering column (37) to obtain said high quality ethanol products (25). Here said first analyzer column (31) is heat integrated with the unused energy in the form of vapours of a part of alcohol vapours (13) from said rectification column (34). Further said second analyzer column (30) is heat integrated with the unused energy in the form of vapours of alcohol (7) from said first analyzer column (31). Further said first analyzer column (31) is heat integrated with the unused energy in the form of vapours of dehydrated alcohol (18) from said dehydration unit (35). Further said high boiler column (36) is heat integrated with the unused energy in the form of vapours of alcohol (19) from said low boiler column (33). Further again said simmering column (37) is heat integrated with the unused energy in the form of vapours of alcohol (20) from said high boiler column (36). The fresh energy in the form of steam is given to units (33), (34) and (35) from an external heat source (29). The heat integration of unused energy is said process is done using reboilers (38). Reboilers are a type of heat exchangers that can exchange heat from a high energy liquid or vapours to low energy liquids. In the disclosed process the columns 30, 31, 32 and 37 are under desired vacuum, while columns 33 and 34 are above atmospheric pressure; and column 36 is at atmospheric pressure. Technical grade alcohol products [TA] are obtained from columns 32, 33, 36 and 37.

[0018] Examples provided below give wider utility of the invention without any limitations as to the variations that may be appreciated by a person skilled in the art. A non-limiting summary of various embodiments is given in the examples and tables, which demonstrate the advantageous and novel aspects of the process disclosed herein. Particular examples of processes in accordance with this invention will now be described with reference to the accompanying drawings

EXAMPLE 1

[0019] In one embodiment of the present invention about 1000 kilolitres of fermented wash

produced by yeast from a carbohydrate containing feedstock was taken, which contained about 10% v/v of ethanol. This fermented wash was split to two unequal feed streams of about 55% and 45% of initial amounts of the wash. The larger feed stream was preheated to about 76 °C by a plate type of heat exchanger receiving unused energy in the form of liquid obtained from processes downstream of analyzer columns, and fed into a first analyzer column operating at a higher pressure receiving unused energy of the rectification column downstream. The smaller feed stream was preheated to about 49 °C by a plate type of heat exchanger receiving unused energy in the form of liquid from processes downstream, and fed into a second analyzer column operating at a lower pressure. Before feeding these streams to the analyzer columns, the stream were passed through degassing column located above the analyzer column in order to degas said feed streams before entry to said analyzer columns. The distilled out ethanol streams from each column were combined and formed a first hydrous ethanol stream. This split primary distillation produced typically about 55% v/v ethanol content in said first hydrous ethanol stream. The second analyzer column was heat integrated by energy in the form of vapours left unused in said first analyzer column operating at higher pressure than said second analyzer column, which operated at a low vacuum condition. In the next step, said a part of first hydrous ethanol stream said second analyzer column was subjected to an aldehyde column, which received unused energy in the form of vapours from said first analyser column. In this aldehyde distillation, aldehyde congeners of said first hydrous ethanol stream were removed leading to the formation of an aldehyde free ethanol stream. In the next step, said aldehyde free ethanol stream along with the vapour condensate from both the analyzer columns was diluted to about 1:3 ethanol to water dilution ratio on volume basis and subjected to a low boiler column, which received fresh energy in the form of steam at need and dilution water left from the rectification column present downstream was used as dilution water. Further said aldehyde free ethanol stream was preheated to about 92 °C by a heat exchanger receiving unused energy left in the rectification lees. In this low boiler distillation, low boiling congeners of said aldehyde free ethanol stream were removed leading to the formation of a second hydrous ethanol stream. In further step, said second hydrous ethanol stream was subjected to a rectification column, which received fresh energy in the form of steam at need. Here, said second hydrous ethanol stream was preheated to about 108 °C by a heat exchanger receiving unused energy left in the rectification lees. In this rectification distillation, the alcohol content of said second hydrous ethanol stream was increased up to 93% v/v of ethanol to get a rectified ethanol stream. In the next step, said rectified ethanol stream [which is in vapour form] was subjected to a dehydration unit, which received fresh energy in the form of steam at need. By dehydration the ethanol content of said rectified ethanol stream was increased to more than 99% v/v of ethanol to obtain an anhydrous ethanol stream. In further step, said anhydrous ethanol stream was subjected to a high boiler column, which received unused energy in the form of vapours from said low boiler column. In this high boiler distillation, high boiling congeners of said anhydrous ethanol stream were removed leading to the formation of a final anhydrous ethanol stream. Then said final anhydrous ethanol stream was subjected to a simmering column to obtain finer grades of ethanol product having improved congener profiles. The said final anhydrous ethanol stream formed an ethanol product called high quality ethanol [HQE] of no odour or taste of any of congeners and suitable for various application like making potable grade ethanol, perfumery and pharmaceutical grade ethanol

with other alcohol products. Also different streams of impure ethanol streams were produced having high ethanol as well as high congener content from aldehyde column, low boiler column, high boiler column and simmering column, which were termed technical ethanol streams and subjected to the preparation of technical alcohol products. Finally about 95 kilolitres of HQNS was obtained from about 1000 kilolitres of fermented wash with 10% v/v of ethanol with consumption of about 2 kilograms of steam per litre of HQE.

EXAMPLE 2

[0020] In another embodiment of the present invention about 1000 kilolitres of fermented wash produced by yeast from a carbohydrate containing feedstock was taken, which contained about 6% v/v of ethanol. This fermented wash was split to two unequal feed streams of about 55% and 45% of initial amounts of the wash. The larger feed stream was preheated to about 76 °C by a plate type of heat exchanger receiving unused energy in the form of liquid obtained from processes downstream of analyzer columns, and fed into a first analyzer column operating at a higher pressure receiving unused energy of the rectification column downstream. The smaller feed stream was preheated to about 49 °C by a plate type of heat exchanger receiving unused energy in the form of liquid from processes downstream, and fed into a second analyzer column operating at a lower pressure. Before feeding these streams to the analyzer columns, the streams were passed through degassing column located above the analyzer column in order to degas said feed streams before entry to said analyzer columns. The distilled out ethanol streams from each column were combined and formed a first hydrous ethanol stream. This split primary distillation produced typically about 43% v/v ethanol content in said first hydrous ethanol stream. The second analyzer column was heat integrated by energy in the form of vapours left unused in said first analyzer column operating at higher pressure than said second analyzer column, which operated at a low vacuum condition. In the next step, said a part of first hydrous ethanol stream was subjected to an aldehyde column, which received unused energy in the form of vapours from said first analyser column. In this aldehyde distillation, aldehyde congeners of said first hydrous ethanol stream were removed leading to the formation of an aldehyde free ethanol stream. In the next step, said aldehyde free ethanol stream along with the vapour condensate from both the analyzer columns was diluted to about 1:3 ethanol to water dilution ratio on volume basis and subjected to a low boiler column, which received fresh energy in the form of steam at need and dilution water left from the rectification column present downstream was used as dilution water. Further said aldehyde free ethanol stream was preheated to about 92 °C by a heat exchanger receiving unused energy left in the rectification leas. In this low boiler distillation, low boiling congeners of said aldehyde free ethanol stream were removed leading to the formation of a second hydrous ethanol stream. In further step, said second hydrous ethanol stream was subjected to a rectification column, which received fresh energy in the form of steam at need. Here, said second hydrous ethanol stream was preheated to about 108 °C by a heat exchanger receiving unused energy left in the rectification leas. In this rectification distillation, the alcohol content of said second hydrous ethanol stream was increased up to 93% v/v of ethanol to get a rectified ethanol stream. In the next step, said rectified ethanol stream [which is in vapour form] was

subjected to a dehydration unit, which received fresh energy in the form of steam at need. Herein before said dehydration step said rectified ethanol stream was treated with an elemental hydroxide like sodium hydroxide, potassium hydroxide, calcium hydroxide or equivalent other hydroxide to improve the pH of said stream to between pH 7 to pH 11. Thereby the formation of compounds like acetal due to reaction of acetaldehyde and ethanol is diminished. This treatment substantially improved the quality of said stream eradicating any acidity associated with it. By dehydration the ethanol content of said rectified ethanol stream was increased to more than 99% v/v of ethanol to obtain an anhydrous ethanol stream. Further said anhydrous ethanol stream was treated with an inorganic oxidising agent like potassium permanganate, hydrogen peroxide, sodium percarbonate or equivalent chemical [added up to 30 PPM concentration] to remove other organic impurities of higher primary and secondary alcohols like butanol, propanol, pentanol, ketones, aldehydes and other chemicals that adversely affect the organoleptic properties of the final products, leading to substantially improvement in the final quality of said stream. This treatment further improved the UV absorbance spectrum of said stream between 220 nm to 270 nm, qualifying said stream for more stringent specifications on said impurities in the final products. In further step, said anhydrous ethanol stream was subjected to a high boiler column, which received unused energy in the form of vapours from said low boiler column. In this high boiler distillation, high boiling congeners of said anhydrous ethanol stream were removed leading to the formation of a final anhydrous ethanol stream. Then said final anhydrous ethanol stream was subjected to a simmering column to obtain finer grades of ethanol product having improved congener profiles. The said final anhydrous ethanol stream formed an ethanol product called high quality neutral spirit [HQE] of no odour or taste of any of congeners and suitable for various application like making potable grade ethanol with other alcohol products. Also different streams of impure ethanol streams were produced having high ethanol as well as high congener content from aldehyde column, low boiler column, high boiler column and simmering column, which were termed technical ethanol streams and subjected to the preparation of technical alcohol products. Finally about 57 kilolitres of HQNS was obtained from about 1000 kilolitres of fermented wash with 6% v/v of ethanol with consumption of about 2.2 kilograms of steam per litre of HQE.

EXAMPLE 3

[0021] In one embodiment of the present invention about 1000 kilolitres of fermented wash produced by yeast from a carbohydrate containing feedstock was taken, which contained about 14% v/v of ethanol. This fermented wash called feed stream was preheated to about 76 °C by a plate type of heat exchanger receiving unused energy in the form of liquid obtained from processes downstream and fed into a first analyzer column operated by unused energy of the rectification column downstream. Before feeding this streams to the analyzer column, it were passed through degassing column located above the analyzer column in order to degas said feed stream before entry to said analyzer column. The distilled out ethanol stream formed a first hydrous ethanol stream. This primary distillation produced typically about 62% v/v ethanol content in said first hydrous ethanol stream. The second analyzer column was heat

integrated by energy in the form of vapours left unused in said first analyzer column operating. In the next step, said a part of first hydrous ethanol stream was subjected to an aldehyde column, which received unused energy in the form of vapours from said first analyser column. In this aldehyde distillation, aldehyde congeners of said first hydrous ethanol stream were removed leading to the formation of an aldehyde free ethanol stream. In the next step, said aldehyde free ethanol stream along with the vapour condensate from both the analyzer columns was diluted to about 1:3 ethanol to water dilution ratio on volume basis and subjected to a low boiler column, which received fresh energy in the form of steam at need and dilution water left from the rectification column present downstream was used as dilution water. Further said aldehyde free ethanol stream was preheated to about 92 °C by a heat exchanger receiving unused energy left in the rectification lees. In this low boiler distillation, low boiling congeners of said aldehyde free ethanol stream were removed leading to the formation of a second hydrous ethanol stream. In further step, said second hydrous ethanol stream was subjected to a rectification column, which received fresh energy in the form of steam at need. Here, said second hydrous ethanol stream was preheated to about 108 °C by a heat exchanger receiving unused energy left in the rectification lees. In this rectification distillation, the alcohol content of said second hydrous ethanol stream was increased up to 93% v/v of ethanol to get a rectified ethanol stream. Herein said rectified ethanol stream was treated with an elemental hydroxide like sodium hydroxide, potassium hydroxide, calcium hydroxide or equivalent other hydroxide to improve the pH of said stream to between pH 7 to pH 11. This treatment substantially improved the quality of said stream eradicating any acidity associated with it. In the next step, said rectified ethanol stream [which is in vapour form] was subjected to a dehydration unit, which received fresh energy in the form of steam at need. By dehydration the ethanol content of said rectified ethanol stream was increased to more than 99% v/v of ethanol to obtain an anhydrous ethanol stream. In further step, said anhydrous ethanol stream was subjected to a high boiler column, which received unused energy in the form of vapours from said low boiler column. Further said anhydrous ethanol stream was treated with an inorganic oxidising agent like potassium permanganate, hydrogen peroxide, sodium percarbonate or equivalent chemical [added up to 30 PPM concentration] to remove other organic impurities of higher primary and secondary alcohols like butanol, propanol, pentanol, ketones, aldehydes and other chemicals that adversely affect the organoleptic properties of the final products, leading to substantially improvement in the final quality of said stream. This treatment further improved the UV absorbance spectrum said stream of between 220 nm to 270 nm, qualifying said stream for more stringent specifications on said impurities in the final products. In this high boiler distillation, high boiling congeners of said anhydrous ethanol stream were removed leading to the formation of a final anhydrous ethanol stream. Then said final anhydrous ethanol stream was subjected to a simmering column to obtain finer grades of ethanol product having improved congener profiles. The said final anhydrous ethanol stream formed an ethanol product called high quality neutral spirit [HQE] of no odour or taste of any of congeners and suitable for various application like making potable grade ethanol with other alcohol products. Also different streams of impure ethanol streams were produced having high ethanol as well as high congener content from aldehyde column, low boiler column, high boiler column, and simmering column, which were termed technical ethanol streams and subjected to the preparation of technical alcohol products. Finally about 133 kilolitres of HQNS was obtained

from about 1000 kilolitres of fermented was with 14% v/v of ethanol with consumption of less than 2 kilograms of steam per litre of HQE.

EXAMPLE 4

[0022] By the process of present invention, different types and grades of ethanol products were obtained. An illustrative typical chemical composition of HQE is listed in TABLE 1. The amount of different congeners is given in parts per million by volume.

TABLE 1: Chemical composition of HQE

Sr No	Component	Composition
1	Ethanol	93-99.9% V/V
2	Acetaldehyde	Less than 0.1 PPM
3	Methyl acetate	Less than 0.1 PPM
4	Ethyl acetate	Less than 0.1 PPM
5	Methanol	Less than 0.1 PPM
6	2-Butanol	Less than 0.1 PPM
7	N-Propanol	Less than 0.1 PPM
8	Iso-butanol	Less than 0.1 PPM
9	Iso-amyl alcohol (Iso-pentanol)	Less than 0.1 PPM
10	N-Amyl Alcohol	Less than 0.1 PPM
11	Acetic acid	Less than 0.1 PPM
12	Furfural	Less than 0.1 PPM
13	2,3 Pentanedione	Less than 0.001 PPM
14	2,3 Butanedione	Less than 0.001 PPM
15	Acetal	Less than 0.1 PPM
16	Isopropanol	Less than 0.1 PPM
17	Dimethyl disulphide	Less than 0.1 PPM
18	Water	Balance

[0023] While the invention has been particularly shown and described with reference to embodiments listed in examples, it will be appreciated that several of the above disclosed and other features and functions, or alternatives thereof, may be desirably combined into many other different systems or applications. Also that various presently unforeseen and unanticipated alternatives, modifications, variations, or improvements therein may be subsequently made by those skilled in the art which are also intended to be encompassed by the following claims. Although the invention has been described with reference to specific preferred embodiments, it is not intended to be limited thereto, rather those having ordinary

skill in the art will recognize that variations and modifications may be made therein which are within the spirit of the invention and within the scope of the claims.

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- [US4784868A \[0007\]](#)
- [US5035776A \[0008\]](#)

PATENTKRAV

1. Destillationsfremgangsmåde til separering af ethanol fra et fortyndet ethanolholdigt udgangsmateriale, omfattende:

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(a) opdeling af udgangsmaterialet i to tilførselsstrømme med uens størrelse;

(b) forvarmning af såvel den større som den mindre af de nævnte tilførselsstrømme;

(c) indføring af de forvarmede tilførselsstrømme parallelt i et par analysekolonner;

10 (d) opretholdelse af et højere tryk i første analysekolonne, som modtager den større tilførselsstrøm, end i anden analysekolonne, som modtager den mindre tilførselsstrøm;

(e) udtrækning af første vandige ethanolstrømme fra den øvre del af analyse-søjlerne og fremføring af disse til en aldehydkolonne for at udskille en aldehydfri ethanolstrøm ved bunddelen og en teknisk alkoholstrøm ved den øvre del af kolonnen;

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(f) iblanding af imellem omkring 3 til omkring 12 dele vand eller brugt bundfald til én del ethanol i den aldehydfrie ethanolstrøm, forvarmningen af denne og underkastning af denne for en lavkogende kolonne for at udskille en anden vandig ethanolstrøm fra den nedre del og en teknisk alkoholstrøm fra den øvre del af kolonnen;

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(g) forvarmning af den anden vandige ethanolstrøm og underkastning af denne for en rektifikationskolonne for at tilvejebringe en rektificeret ethanolstrøm, hvor den rektificerede ethanolstrøm behandles med et elementært hydroxid for at justere dennes pH til mellem pH 7 og pH 11;

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(h) dehydrering af den rektificerede ethanolstrøm under anvendelse af en dehydreringsenhed for tilvejebringelse af en vandfri ethanolstrøm, hvor dehydreringen af den rektificerede ethanolstrøm opnås ved hjælp af en membranseparationsteknik, molekylsigte-dehydrering eller adsorptionsseparation af vand eller en kombination deraf;

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(i) indføring af den vandfrie ethanolstrøm til en højerekogende kolonne for udskillelse af en teknisk alkoholstrøm ved bunddelen og en endelig vandfri ethanolstrøm ved den øvre del af kolonnen, hvor den vandfrie ethanolstrøm behandles med et uorganisk oxidationsmiddel op til 30 ppm koncentration og

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(j) indføring af den endelige vandfrie ethanolstrøm til en kogekolonne for opnåelse af ethanolproduktstrømme ved eventuel tilføjelse af vand til den endelige vandfrie ethanolstrøm i forskellige mængder.

2. Fremgangsmåde ifølge krav 1, hvor den større tilførselsstrøm omfatter imellem 50 til 70 vægt-% af det totale udgangsmateriale.
3. Fremgangsmåde ifølge krav 1, hvor aldehydkolonnen udskiller i det mindste én aldehydkomponent, som foreligger i de første vandige ethanolstrømme, som opnås fra analysekolonnerne.
4. Fremgangsmåde ifølge krav 1, hvor den lavtkogende kolonne udskiller i det mindste én lavt kogende komponent, som foreligger i den aldehydfrie ethanolstrøm, som opnås fra aldehydkolonnen.
5. Fremgangsmåde ifølge krav 1, hvor rektifikationskolonnen yderligere oprenser den anden vandige ethanolstrøm, som opnås fra den lavtkogende kolonne for i det mindste én højt kogende eller én lavtkogende komponent eller begge.
6. Fremgangsmåde ifølge krav 1, hvor den højt kogende kolonne udskiller i det mindste én højt kogende komponent foreliggende i den vandfrie ethanolstrøm opnået fra dehydreringsenheden.
7. Fremgangsmåde ifølge krav 1, hvor vand eventuelt tilføjes i sluttrinnet i forskellige mængder for opnåelse af ethanolprodukter med omkring mellem 0,1 til 6 volumen-% vand.
8. Fremgangsmåde ifølge krav 1, hvor ikke mere end omkring 2 kg damp kræves for at opnå omkring 1 liter af et ethanolprodukt.
9. Fremgangsmåde ifølge krav 1, hvor varme, som efterlades ubrugt i rektifikationskolonnen, integreres med den første analysekolonne.
10. Fremgangsmåde ifølge krav 1, hvor varme, som efterlades ubrugt i dehydreringsenheden, integreres med den første analysekolonne.
11. Fremgangsmåde ifølge krav 1, hvor varme, som efterlades ubrugt i den første analysekolonne, integreres med den nævnte aldehydkolonne eller den anden analysekolonne.

12. Fremgangsmåde ifølge krav 1, hvor varmen, som efterlades ubrugt i den lavtkogende kolonne, integreres med den højt kogende kolonne.
13. Fremgangsmåde ifølge krav 1, hvor frisk energi i form af damp tilføres den lavtkogende kolonne, rektifikationskolonnen og dehydreringsenheden efter behov.
14. Fremgangsmåde ifølge krav 1, hvor forvarmningen ved forskellige trin opnås ved integrering af varme, som efterlades ubrugt i andre enhedstrin.
15. Fremgangsmåde ifølge krav 1, hvor det fortyndede ethanolholdige udgangsmateriale omfatter: en fermenteret væske eller syntetisk ethanolstrøm eller en kombination deraf.
16. Fremgangsmåde ifølge krav 1, hvor mængden af ethanol i det fortyndede ethanolholdige udgangsmateriale er i det mindste 3 volumen-%.
17. Fremgangsmåde ifølge krav 1, hvor de tekniske alkoholstrømme opnås fra aldehydkolonnen, den lavtkogende kolonne, den højt kogende kolonne og kogekolonnen.
18. Fremgangsmåde ifølge krav 1, hvor den rektificerede ethanolstrøm opnået fra rektifikationskolonnen indeholder i det mindste 93 volumen-% ethanol.
19. Fremgangsmåde ifølge krav 1, hvor den vandfrie ethanolstrøm opnået fra dehydreringsenheden indeholder i det mindste 99 volumen-% ethanol.
20. Fremgangsmåde ifølge krav 1, hvor ethanolproduktstrømmene opnået fra kogekolonnen ikke omfatter mere end 1 ppm methanol og ikke mere end 1 ppm aldehyd.
21. Fremgangsmåde ifølge krav 1, hvor ethanolproduktstrømmene opnået fra kogekolonnen ikke omfatter mere end 1 ppm ethylacetat og ikke mere end 2 ppm fuselolier.
22. Fremgangsmåde ifølge krav 1, hvor ethanolproduktstrømmene opnået fra kogekolonnen er neutral i lugt eller smag.
23. Fremgangsmåde ifølge krav 1, hvor, i de første vandige ethanolstrømme opnået fra analysekolonnerne, mængden af isopropylalkohol er reduceret til i det mindste 40% af startmængden foreliggende i tilførselsstrømmene.

24. Destillationsapparat til udvinding af ethanol fra et fortyndet ethanolholdigt udgangsmateriale, til anvendelse ved en fremgangsmåde ifølge ethvert af kravene 1 til 23, omfattende:

- 5 (a) en udgangsmateriale opsplittingsenhed til at opnå to tilførselsstrømme med uens størrelse;
- (b) en forvarmingsenhed til opvarmning af såvel den større som den mindre af de nævnte tilførselsstrømme;
- (c) en første analysekolonne og en anden analysekolonne, en aldehydkolonne, en 10 lavtkogende kolonne, en rektifikationskolonne, en dehydreringsenhed, en højt-kogende kolonne og en kogekolonne;
- (d) en tilvejebringelse til indføring af den større af tilførselsstrømmene til den første analysekolonne og tilvejebringelse til indføring af den mindre af tilførselsstrømmene til den anden analysekolonne;
- 15 (e) en første reboiler til kondensering af alkoholdampe fra dehydreringsenheden nedstrøms, for tilførsel af varmen, som kræves i den første analysekolonne;
- (f) en anden reboiler til kondensering af overskydende dampe fra rektifikationskolonnen nedstrøms for analysekolonnerne, for tilførsel af varmen, som kræves i den første analysekolonne;
- 20 (g) en tredje reboiler til kondensering af overskydende dampe fra den første analysekolonne, for tilførsel af varmen, som kræves i den anden analysekolonne;
- (h) en tilvejebringelse til udtrækning af første vandige ethanolstrømme fra en øvre del af analysekolonnerne;
- (i) en tilvejebringelse til forvarmning og indføring af de første vandige ethanolstrømme til aldehydkolonnen;
- 25 (j) en tilvejebringelse til udtrækning af en aldehydfri ethanolstrøm fra en bunddel af aldehydkolonnen;
- (k) en fortyndingsbeholder til fortynding af den aldehydfrie ethanolstrøm med vand eller brugt bundfald;
- 30 (l) en tilvejebringelse til forvarmning og indføring af den fortyndede aldehydfrie ethanolstrøm i rektifikationskolonnen;
- (m) en tilvejebringelse til udtrækning af en anden vandig ethanolstrøm fra den nedre del og til udtrækning af en teknisk alkoholstrøm fra den øvre del af den lavtkogende kolonne;
- 35 (n) en tilvejebringelse til forvarmning og indføring af den anden vandige ethanolstrøm i rektifikationskolonnen;

- (o) en tilvejebringelse til udtrækning af en rektificeret ethanolstrøm fra en øvre del af rektifikationskolonnen;
- (p) en tilvejebringelse til indføring af et elementært hydroxid i den rektificerede ethanolstrøm for justering af dennes pH til imellem 7 og 11;
- 5 (q) en tilvejebringelse til indføring af den rektificerede ethanolstrøm i dehydreringsenheden, hvor dehydreringsenheden er én eller flere af en membranseparationsenhed, en molekylsigteenhed eller en adsorptionsenhed;
- (r) en tilvejebringelse til udtrækning af en vandfri ethanolstrøm fra dehydreringsenheden;
- 10 (s) en tilvejebringelse til indføring af den vandfrie ethanolstrøm i den højt kogende kolonne;
- (t) en tilvejebringelse til indføring af et uorganisk oxidationsmiddel i den vandfrie ethanolstrøm;
- (u) en tilvejebringelse til udskillelse af en teknisk alkoholstrøm ved bunddelen og en endelig vandfri ethanolstrøm ved den øvre del af den højt kogende kolonne;
- 15 (v) en tilvejebringelse til indføring af den vandfrie ethanolstrøm i en kogekolonne med organer til eventuelt at iblande vand; og
- (w) en tilvejebringelse til udtagning af ethanolproduktstrømme fra kogekolonnen.
- 20 25. Apparaturløse ifølge krav 24, hvor varme, som efterlades ubrugt i den lavt kogende kolonne, tilføres den højt kogende kolonne.
26. Apparaturløse ifølge krav 24, hvor varme, som efterlades ubrugt i den højt kogende kolonne, tilføres rektifikationskolonnen.
- 25 27. Apparaturløse ifølge krav 24, hvor varme, som efterlades ubrugt i rektifikationskolonnen, tilføres den første analysekolonne.
28. Apparaturløse ifølge krav 24, hvor varme, som efterlades ubrugt i den første analysekolonne, tilføres til den anden analysekolonne og aldehydkolonnen.
- 30 29. Apparaturløse ifølge krav 24, hvor varme, som efterlades ubrugt i dehydreringsenheden, tilføres til den første analysekolonne.
- 35 30. Apparaturløse ifølge krav 24, hvor kogekolonnen indeholder kobberindbygningsdele til effektiv fjernelse af methanol og svovlforurening fra ethanolproduktstrømmene.

31. Apparatur ifølge krav 24, hvor ikke mere end omkring 2 kg damp kræves for at opnå 1 liter ethanolprodukt.

DRAWINGS

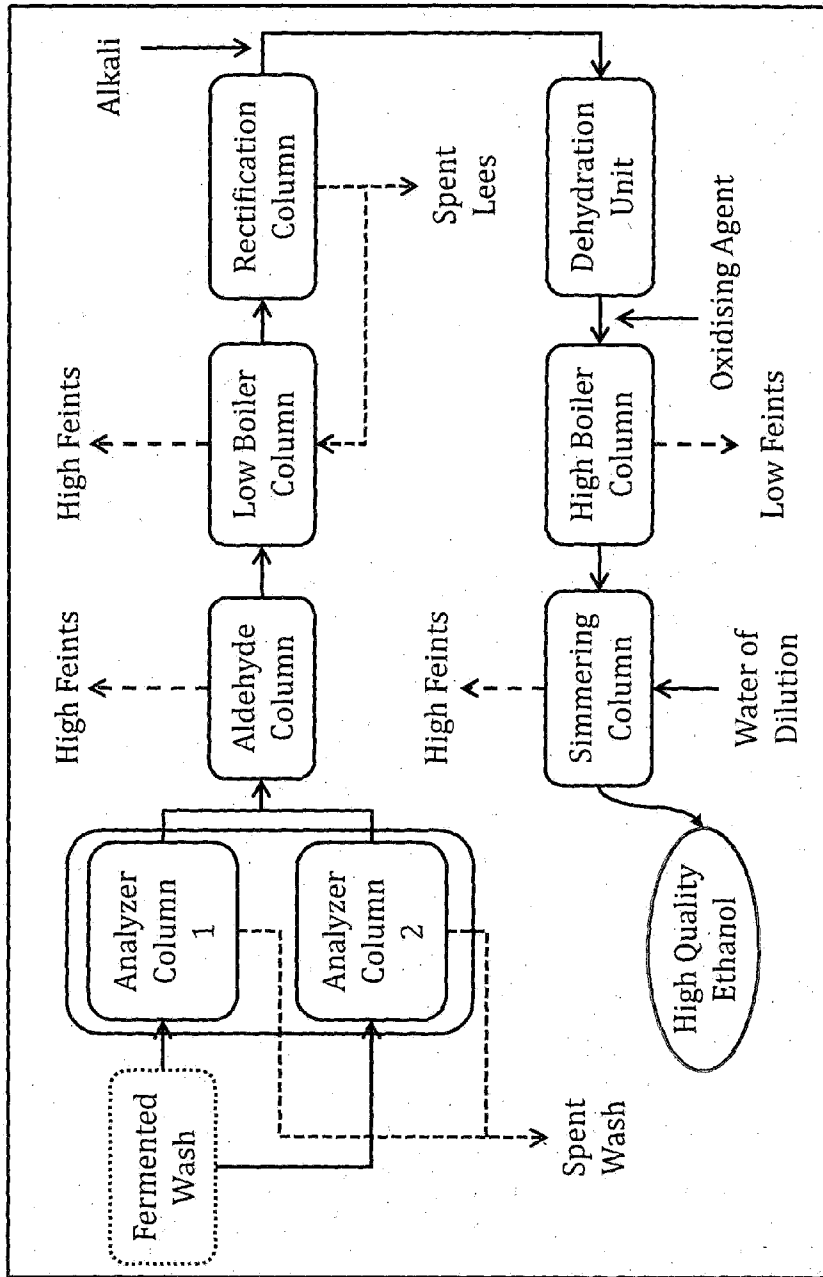


FIGURE 1

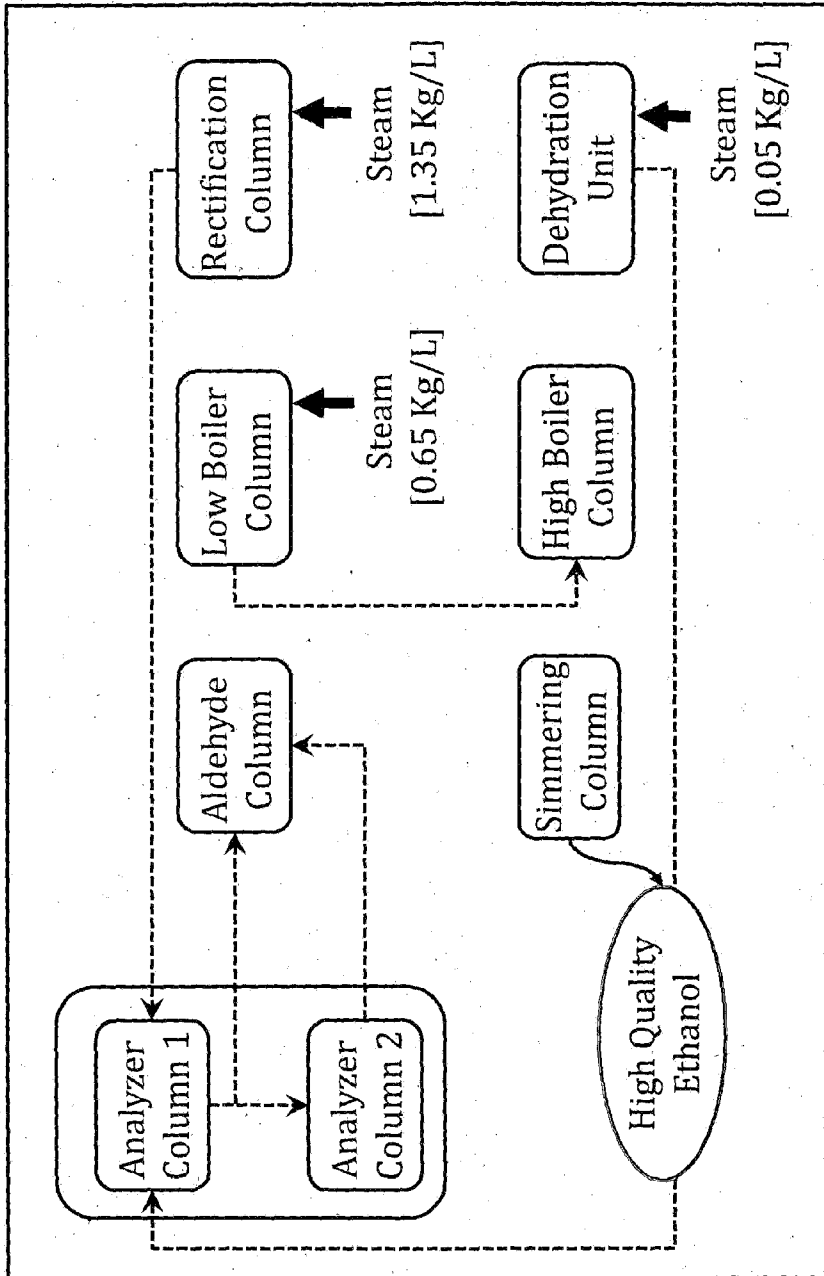


FIGURE 2

