RES

PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Separating Organic Oxygen Compounds from Aqueous Mixtures by Extractive Distillation

We, STANOLIND OIL AND GAS COMPANY, a corporation organised under the laws of the State of Delaware, United States of America, of Stanolind Building, City of Tulsa, State of Oklahoma, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

Our invention relates to a method for separating organic compounds from one another and from complex organic mixtures in which at least some of the components thereof form azeotropes with one or more of the constituents present therein. More particularly, it relates to the separation of ethanol, isopropanol, and methyl ethyl ketone from one another and from complex aqueous mixtures containing these and other organic compounds.

In accordance with the present invention there is provided a process for the recovery of ethanol from aqueous mixtures containing isopropanol together with at least one other oxygenated organic compound capable of forming an azeotrope with ethanol, which comprises subjecting said mixture to fractional distillation in a column, in which a heat input of from about 100 to about 150 B.t.u./pound of bottoms is employed to give a bottom column temperature of from about 90° to about 98° C. and an overhead temperature of from about 73° to about 88° C., introducing water into said column countercurrently to the flow of the vaporized mixture in an amount such that the concentration of water in the liquid at any point in the column is from about 90 to about 99 mol per cent, and withdrawing an aqueous bottom stream containing the bulk of the ethanol originally present and an overhead fraction containing from about 1 to about 20 weight per cent of ethanol, based on the concentration thereof in the original mixture, together with isopropanol and at least one other oxygenated organic compound capable of forming an azeotrope with ethanol.

The present invention further provides a process for the separate recovery of ethanol and isopropanol from an aqueous mixture containing butyraldehyde, ethyl acetate, and methyl ethyl ketone, which process comprises subjecting said mixture to fractional distillation in a column in which a heat input of from 80 to 120 B.t.u/pound of bottoms is employed, introducing water into said column countercurrently to the flow of the vaporized mixture in an amount such that the concentration of water in the liquid at any point in the column is from about 90 to about 99 mol per cent and withdrawing a bottoms stream containing methyl ethyl ketone, ethanol, and isopropanol and an overhead fraction comprising butyraldehyde and ethyl acetate.

Mixtures of the type contemplated by our invention are encountered in the water soluble chemicals fraction obtained in the now generally known hydrocarbon synthesis process involving the reduction of carbon monoxide with hydrogen in the presence of a fluidized iron catalyst. In the hydrocarbon synthesis plants now designed for commercial purposes, having capacities of the order of 6000 barrels of liquid hydrocarbons per day, the chemicals present in mixtures of the type contemplated by the process of our invention constitute approximately 30 per cent of the total water soluble chemicals produced or about 140,000 pounds per day. Accordingly, for economic and other reasons, it is essential that these chemicals be recovered, each in as highly purified form as possible.

Mixtures typical of those with which we are concerned and the relative concentrations in which each component thereof is present are indicated below;—

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Component	Wt. %
Butyraldehyde - - -	4.0
Ethyl acetate - - - -	1.4
Methyl ethyl ketone - -	11.1
Methyl propyl ketone - -	1.7
Ethanol - - - - - -	74.0
Isopropanol - - - -	1.4
Water - - - - - -	6.3
Other impurities - - -	0.1

Some appreciation of the difficulties encountered in attempting to obtain any or all of the above-mentioned oxygenated organic compounds in a substantially pure form when present in such mixtures may be realized by reference to the boiling points of the pure compounds, as well as to their azeotropes either in the aqueous or anhydrous state.

Boiling points of pure chemicals	B.P. °C.
Butyraldehyde - - - -	75.7
Ethyl acetate - - - - -	77.1
Ethanol - - - - - -	78.4
Methyl ethyl ketone - -	79.6
Isopropanol - - - -	82.4
Methyl propyl ketone - -	102.0

Boiling points of azeotropes	B.P. °C.
Butyraldehyde - ethanol - water - - - - - -	67.2
Butyraldehyde-water - -	68.0
Ethanol-ethyl acetate-water	70.3
Ethyl acetate-water - -	70.4
Ethyl acetate-ethanol - -	71.8
Ethanol - methyl ethyl ketone-water - - -	73.2
Methyl ethyl ketone-water	73.5
Ethanol - methyl ethyl ketone - - - - - -	74.8
Ethyl acetate-methyl ethyl ketone - - - - -	76.7
Ethanol - methyl propyl ketone - - - - - -	77.7
Ethanol - methyl propyl ketone-water - - -	78.1
Ethanol-water - - -	78.2
Isopropanol-water - -	80.4
Methyl propyl ketone-water	82.9

It will be readily apparent to one skilled in the art to which the invention relates, that the separation in substantially pure form of even a single component from aqueous mixture of the above type presents a formidable recovery problem. Even if water were removed from this mixture by one of several methods, fractionation of the anhydrous chemicals to yield pure products would not be practicable.

It is an object of the invention to provide an efficient method for the separation of ethanol, isopropanol, methyl ethyl ketone, and methyl propyl ketone from complex mixtures of the type referred to above. It is a further object of our invention to provide a process for obtaining highly purified ethanol from mixtures of the aforesaid type, which meets A.C.S. purity requirements with respect to isopropanol, i.e., not more than 30 parts of isopropanol per million parts of ethanol.

We have now discovered that the foregoing and other objects can be accomplished by subjecting crude mixtures of the type described above to a series of extractive distillations with water under controlled conditions of water concentration and heat input described hereinbelow. According to the process of the invention, a crude mixture of the aforesaid type is extractively distilled with water, whereby the bulk of the ethanol is washed down the column in the form of a dilute aqueous solution. In this step the fractionating tower is operated under conditions such that an aqueous overhead fraction is secured which contains butraldehyde, ethyl acetate, methyl ethyl ketone, methyl propyl ketone, isopropanol, and some ethanol. The most difficult component to separate from the ethanol is isopropanol, and we have found that by taking overhead a relatively small percentage of the ethanol at this point in the process the isopropanol in the bottoms ethanol can be reduced to about 30 parts per million or less. We have found that in order to effect a satisfactory separation, dilution water should be added to the column in an amount such that the concentration of water in the liquid within the column is from about 90 to 99 mol per cent, preferably in the range of 95 mol per cent. The amount of net distillate drawoff is carefully controlled so that ethanol of the desired purity is produced. The amount of heat supplied to the column is normally in the range of 100 to 150 B.t.u./pound of bottoms. A minimum amount of heat is required to secure satisfactory stripping of isopropanol and traces of other impurities, especially unsaturated components which deleteriously affect purity of the ethanol. The use of excessive amounts of heat beyond the stripping requirement impairs the efficiency of the column due primarily to decreasing water concentration within the column. The temperatures employed may vary widely, but for the majority of feeds we have found that the tower bottom temperatures range from about 90—98° C., preferably about 96° C, (corrected to atmospheric pressure) and the overhead temperatures range from about 73—88° C, preferably about 76° C.

The quantity of ethanol withdrawn in the overhead stream will vary with the composition of the original feed and the efficiency of the fractionating column employed. Thus, with feeds of the type encountered in the separation of the non-acid water soluble chemicals obtained in the conventional hydrocarbon synthesis process, the ethanol content will generally be found to be in the neighborhood of 60 to 80 per cent of the total chemicals present, while isopropanol is generally present in a concentration of from about 1 to 3 per cent. Accordingly, we have found that with mixtures of the aforesaid type a satisfactory separation of the isopropanol from the bulk of the ethanol may be effected by taking overhead in the initial distillation step a quantity of ethanol corresponding to from about 1 to about 10 weight per cent of that in the original feed mixture. This, for example, with a fractionating column having 65 plates and operating at an efficiency of 50 to 60 per cent, we have observed that by taking overhead only about 1.3 weight per cent of the ethanol originally present in the feed, a highly purified form of ethanol (containing about 10 parts per million of isopropanol) can be obtained. With more efficient fractionating columns the amount of ethanol required to be taken overhead becomes less than 1 weight per cent. However, as the concentration of ethanol in the feed decreases or the relative percentage of isopropanol in the feed increases, (with a given column and other operating conditions remaining constant) it becomes necessary to withdraw higher percentage of ethanol in the overhead in order to insure a substantially complete separation of the isopropanol from the ethanol in the bottoms. For example, with feeds in which the ratio of ethanol to isopropanol is of the order of 10 to 1 instead of about 30 to 1, as is generally the proportion in the case of hydrocarbon synthesis water-soluble-chemicals mixtures, the amount of ethanol withdrawn in the overhead may correspond to as much as about 20 weight per cent of that present in the original feed in order that a satisfactory removal of isopropanol may be achieved.

The dilute aqueous ethanol solution from the base of the column is sent to a conventional fractionating column wherein a constant boiling mixture of ethanol and water of high purity is removed as distillate. The bottoms, which is essentially water, is recycled to provide dilution water for the extractive distillation operation described above. A portion of this water is continuously rejected to the sewer to provide a purge for any heavy impurities in the system.

The crude distillate from the above extractive distillation operation is then subjected to a second extractive distillation with water whereby methyl ethyl ketone, methyl propyl ketone, ethanol, and isopropanol are washed down the column and an aqueous mixture of butyraldehyde and ethyl acetate is taken overhead as distillate.

In general, it may be said that in order to effect a satisfactory separation of butyraldehyde and ethyl acetate from methyl ethyl ketone, methyl propyl ketone, ethanol, and isopropanol at this point, the dilution water should be added to the column in an amount such that the concentration of water in the liquid at any point in the column is from about 90 to 99 mol per cent and preferably about 95 mol per cent. The amount of net overhead withdrawn should be carefully controlled to avoid forcing methyl ethyl ketone, ethanol, or isopropanol overhead or butyraldehyde and ethyl acetate into the bottoms at this stage. Ordinarily the heat input should be in the range of 80—120 B.t.u./pound of bottoms, preferably about 90 B.t.u./pound of bottoms. It has been our observation, however, that the use of an excessive amount of heat impairs the efficiency of the column since under such conditions the concentration of water in the column falls below the value we have found suitable for effecting the desired separation. In this connection, the temperatures employed may vary rather widely. For the majority of feeds, however, we have found that bottom tower temperatures of from 82° to about 94° C. (corrected to atmospheric pressure), preferably about 85° C. and top tower temperatures of from about 68° to about 77° C, preferably 71° C, are satisfactory. The overhead stream obtained in this operation is an aqueous mixture of butyraldehyde and ethyl acetate which may, if desired, be further separated into its individual components. The bottoms portion obtained as a result of this distillation step contains ethanol, isopropanol, methyl ethyl ketone, and methyl propyl ketone in a total concentration of the order of 1 mol per cent. The dilute bottoms fraction containing these chemicals is introduced at an intermediate point in another fractionating column where a third extractive distillation operation is effected. In this separation step, however, the water azeotropes of methyl ethyl ketone and methyl propyl ketone free of alcohols are collected as an overhead stream, a dilute (from about 2 to 10 weight per cent) aqueous solution of isopropanol and ethanol is withdrawn from the column as a side stream below the point at which the feed is introduced, while water is withdrawn from the bottom of the column and recycled to the previous column. In effecting this separation dilution water is introduced at the top of the column in an amount such that the concentration of water in the liquid at any point in the column above the point of side stream withdrawal ranges between about 80 and 99 mol per cent, preferably between about 95 and 99 mol per cent. If the concentration of water above the point of side stream withdrawal is between 80 and 99 mol per cent, the concentration of water at any level below the point of sidestream withdrawal would necessarily be above 80 mol per cent. In instances where it is not considered necessary or desirable to effect a sharp separation of methyl propyl ketone from ethanol and isopropyl alcohol, the minimum concentration of water in the column may be lower than 95 mol per cent and still result in a satisfactory separation of the methyl ethyl ketone from the aforesaid alcohols. Thus, in mixtures of the type here involved and which contain varying amounts of methyl propyl ketone, a substantially complete separation of methyl ethyl ketone from the remaining components of the mixture can be accomplished through the use of as little as about 70 mol per cent water in the column. Heat input to the column will normally be between 125—300 B.t.u./pound of bottoms. Under these conditions isopropanol and ethanol relatively free from ketones can be withdrawn in the form of an aqueous solution as a side stream below the point at which the feed is introduced. The observations relative to heat input discussed above are equally pertinent to the separation of methyl ethyl ketone, methyl propyl ketone, and water from isopropanol, ethanol, and water. While the actual temperatures employed at this stage of the process may vary widely, we have found that in the majority of instances the bottom temperatures will be the boiling point of water, while top temperatures may be in the range of from about 73° to 83° C. and preferably about 74° C. The temperature at which the side stream of aqueous isopropanol and ethanol is withdrawn should generally range between about 92° and about 99° C. (corrected to atmospheric pressure) depending on the concentration selected for the drawoff stream.

If desired, the methyl ethyl ketone and methyl propyl ketone contained in the distillate from this operation can be separated by first removing the azeotropic water and then separating the anhydrous ketones by means of conventional fractional distillation.

The dilute aqueous side stream of isopropanol and ethanol is next introduced at an intermediate point of still another extractive distillation column where isopropanol is taken off overhead and ethanol is withdrawn as a bottoms stream. The isopropanol is removed in the form of an azeotrope with water by introducing the dilution water at a point substantially below the take-off level for the aqueous isopropanol. In this distillation the quantity of water introduced is such that the concentration in the liquid at any point in the column below the point of water introduction is in the range of from about 90 to 99 mol per cent, preferably 95 mol per cent. The aqueous ethanol solution obtained from the base of this column is recycled to supply a portion of the dilution water for the column initially described in the process of the invention. Bottoms temperatures may range from 90° to 98° C. (corrected to atmospheric pressure), but are generally preferably held to about 97° C, while the top tower temperature is about 80.6° C. corresponding to the boiling point of the isopropanol-water azeotrope. Heat input is preferably in the range of 100—150 B.t.u./pound of bottoms.

One embodiment of the invention is illustrated diagrammatically by the accompanying drawing. Referring to the drawing, a charging stock comprising essentially an aqueous solution of butyraldehyde, ethyl acetate, methyl ethyl ketone, methyl propyl ketone, isopropanol, and ethanol obtained, for example, in the hydrogenation of carbon monoxide in the presence of a fluidized iron catalyst, is introduced through line 2 into an intermediate section of column 4 where it is fractionally distilled. The vapors rising through the column encounter a stream of water introduced at the top thereof through line 6 Under these conditions ethanol is selectively washed down the column and withdrawn therefrom through line 8 after which this solution is introduced into column 10 where a constant boiling mixture of ethanol and water is withdrawn overhead through line 12, part of which is returned to the top of the column through line 14 as reflux. The bottoms, which consists essentially of water, is withdrawn through line 16 and a portion thereof transferred through line 18 to the top of column 4 to be recycled as dilution water. The remaining bottoms water is withdrawn through line 17 and run to the sewer. The overhead fraction from column 4 which contains butyraldehyde, ethyl acetate, methyl ethyl ketone, methyl propyl ketone, isopropanol, water, and a relatively small amount (usually from about 1 to 10 weight per cent, based on the concentration of ethanol in the original feed) of ethanol is withdrawn through line 20 and introduced into column 22 where the feed is extractively distilled. The vapours rising in column 22 encounter a stream of water introduced at the top thereof through line 27. In this column butyraldehyde, ethyl acetate, and water are removed in the form of azeotropes as an overhead fraction through line 26 and partially recycled as reflux through line 28. The bottoms portion, which consists of an aqueous solution of methyl ethyl ketone, methyl propyl ketone, isopropanol, and ethanol, is withdrawn through line 30 and introduced into column 32 where extractive distillation is again effected by introducing water into the column through line 35, resulting in methyl ethyl ketone, methyl propyl ketone, and azeotropic water going overhead through line 34, some of which is returned as reflux through line 36, while a side stream of isopropanol, ethanol, and water is withdrawn through line 38 and introduced into column 40. The bottoms from column 32, consisting essentially of water, is withdrawn through line 33 and in part recycled as dilution water to column 22 through line 24 while the remainder is withdrawn through line 51 and run to the sewer. Isopropanol is separated from ethanol in column 40 by means of still another extractive distillation with water which is introduced through line 42. In this instance an azeotrope of isopropanol and water is withdrawn through line 44, part of which is returned to column 40 through line 46 as reflux, while a dilute solution of ethanol is withdrawn as a bottom stream through line 48 and recycled through line 50 to column 4, where it is employed as a source of dilution water.

In the above-mentioned drawing reference to certain equipment such as pumps, reboilers, gages and the like which obviously would be necessary to operate the process has been intentionally omitted. Only sufficient equipment has been diagrammatically shown to illustrate the process.

In every instance where separation is effected by means of extractive distillation, the amount of heat which may be admitted to the column employed is primarily determined by the feed composition, the amount of dilution water added, and the type of separation desired. In contrast to conventional fractionating columns, however, the heat input must be established between definite maximum and minimum limits if, as hereinbefore defined, successful operation is to be obtained. While our process may be carried out at reduced or superatmospheric pressures, we prefer, in general, to employ atmospheric pressure.

Among the outstanding and surprising features of the invention is the fact that by the use of only water as an extractive distillation agent, sharp separations can be made between the several constituents which not only boil very close together but form numerous azeotropes. This fact appears to be all the more surprising when it is realized that the concentration of water employed in such extractive distillation processes is generally within the relatively narrow range of 90—95 mol per cent. For example, in the first extractive distillation operation only ethanol is removed from the base of the column while all other constituents, including those which in the anhydrous state boil as much as 25° C. higher than ethanol, are removed overhead. Even under the conditions specified for this column, the separation of isopropanol from ethanol is very difficult, and we have found it necessary to withdraw some of the ethanol in the distillate in order to effect substantially complete removal of isopropanol.

By changing conditions slightly in the second extractive distillation, the alcohols and ketones are separated from the aldehyde and ester with which they form close boiling azeotropes. In the third extractive distillation, both alcohols are separated from both ketones by taking off the latter as an overhead stream, in spite of the fact that the heavy ketone boils considerably higher than either of the alcohols, and that numerous azeotropes are possible. In the fourth extractive distillation, by employing high dilution and precise temperature control, it is possible to achieve a sharp separation between the two most difficultly separable constituents, namely isopropanol and ethanol. Thus, it is our observation that the components of the mixtures involved show varying degrees of non-ideality in the presence of water and by careful control of such variables as heat input, water concentration, and temperature gradient it is possible to effect the separations herein described.

It will be apparent, in view of the foregoing description, that the process of the invention may be modified in numerous ways without materially departing from the scope thereof. Thus, while the application of the invention has been described principally in connection with the separation of components present in the proportions typical of those found in the water soluble chemicals stream obtained in the hydrocarbon synthesis process, it is to be strictly understood that the invention is equally applicable to the treatment of mixtures in which the common components of the aforesaid water soluble chemicals stream are to be found in proportions substantially different.

The temperature values given throughout this specification refer to operation at atmospheric pressure or are corrected to such pressure.

Patent Specification No. 647,903 describes and claims a process for the separation of at least two water-soluble alcohols of differing molecular weights, which produce close boiling aqueous azeotropes, from feed mixtures containing them, with or without corresponding carbonyl compounds, which comprises continuously introducing the feed stock into a fractionating column at an intermediate point thereof, maintaining the portion of the column above the feed inlet as a rectification zone, continuously introducing water into the top of the said column in sufficient quantity to produce an internal reflux in the said rectification zone containing at least 65 mol per cent water and sufficient to alter the relative volatilities of the components of the feed mixture to produce an overhead distillate containing the higher molecular weight alcohol or alcohols and any carbonyl compounds present, and a bottom stream comprising a dilute aqueous solution of the lowest molecular weight alcohol, maintaining the portion of the column below the feed inlet as a stripping zone in which the dilute solution of the alcohol formed as the bottoms in the rectification zone is reboiled and refluxed, continuously removing from the top of said column a distillate containing the higher molecular weight alcohol or alcohols with any carbonyl compounds, and continously removing from the bottom of the said column a substantially pure dilute aqueous solution of the lowest molecular weight alcohol.

What we claim is:—

1. A process for the recovery of ethanol from aqueous mixtures containing isopropanol together with at least one other oxygenated organic compound capable of forming an azeotrope with ethanol, which comprises subjecting said mixture to fractional distillation in a column, in which a heat input of from about 100 to about 150 B.t.u./pound of bottoms is employed to give a bottom column temperature of from about 90° to about 98° C. and an overhead temperature of from about 73° to about 88° C. introducing water into said column countercurrently to the flow of the vaporized mixture in an amount such that the concentration of water in the liquid at any point in the column is from about 90 to about 99 mol per cent, and withdrawing an aqueous bottom stream containing the bulk of the ethanol originally present and an overhead fraction containing from about 1 to about 20 weight per cent of ethanol, based on the concentration thereof in the original mixture, together with isopropanol and at least one other oxygenated organic compound capable of forming an azeotrope with ethanol.

2. A process according to Claim 1, in which the ethanol content of the feed mixture is between about 60 and about 80 weight per cent and the amount of ethanol taken overhead is from about 1 to about 10 weight per cent, based on the quantity of ethanol in said feed mixture.

3. A process for the separate recovery of ethanol and isopropanol from aqueous mixture containing butyraldehyde, ethyl acetate, and methyl ethyl ketone, such as, for example, the overhead fraction of Claim 1 or 2, which process comprises subjecting said mixture to fractional distillation in a column in which a heat input of from 80 to 120 B.t.u./pound of bottoms is employed, introducing water into said column countercurrently to the flow of the vaporized mixture in an amount such that the concentration of water in the liquid at any point in the column is from about 90 to about 99 mol per cent, and withdrawing a bottoms stream containing methyl ethyl ketone, ethanol, and isopropanol and an overhead fraction comprising butyraldehyde and ethyl acetate.

4. A process according to Claim 3, in which the aqueous mixtures of oxygenated organic compounds are of the type produced in the synthesis of hydrocarbons from carbon monoxide and hydrogen, said fractional distillation is carried out at a bottom tower temperature of from 82° to about 94° C. and an overhead temperature of from about 68° to about 77° C.

5. A process for the separate recovery of ethanol and isopropanol from aqueous mixtures containing methyl ethyl ketone, ethanol, and isopropanol, such as, for example, the bottoms stream of claim 3 or 4, which process comprises subjecting said mixture to fractional distillation in a column in which a heat input of 125 to 300 B.t.u./pound of bottoms is employed, introducing water into said column counter-currently to the flow of the vaporized mixture in an amount such that the concentration of water in the liquid at any point in the column is from about 70 to about 99 mol per cent, and withdrawing an overhead fraction at a temperature of from about 73° to 83° C. containing methyl ethyl ketone and water and a side stream containing ethanol, isopropanol, and water.

6. A process according to Claim 5 of

treating the bottoms stream of Claim 3 or 4, which stream also contains methyl propyl ketone, in which the water concentration in the liquid at any point in the column is from about 95 to about 99 mol per cent, and an overhead fraction containing methyl ethyl ketone, methyl propyl ketone, and water is withdrawn at a temperature of from about 73° to 83° C. and a side stream containing ethanol, isopropanol, and water is withdrawn at a temperature of from about 92° to 99° C.

7. A process according to Claim 5 or 6, which comprises thereafter subjecting said side stream to distillation in a column in which a heat input of 100 to 150 B.t.u./pound of bottoms is employed, introducing water into said column at an intermediate point thereof countercurrently to the flow of the vaporized mixture in an amount such that the concentration of water in the liquid at any point in the column below the water inlet is from 90 to about 99 mol per cent to obtain an overhead fraction of aqueous isopropanol and a bottom stream of aqueous ethanol.

8. A process according to Claim 7, in which the water is introduced into said column in an amount such that the concentration of water in the liquid at any point in the column below the water inlet is from about 95 to about 99 mol per cent.

9. A process according to Claim 7 or 8, in which the heat input is sufficient to give a bottom tower temperature of about 90° to about 98° C. and an overhead temperature of from 73° to about 88° C.

10. The new and improved process of recovering highly purified ethanol from complex mixtures of oxygenated organic compounds substantially as herein described.

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684,968 COMPLETE SPECIFICATION

1 SHEET

This drawing is a reproduction of the Original on a reduced scale.