

DESIGN TECHNIQUES USED FOR THE DEVELOPMENT OF AN AZEOTROPIC DISTILLATION PROCESS WHICH USES A BINARY ENTRAINER FOR SEPARATION OF OLEFINS FROM ACIDS AND OTHER OXYGENATES

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ABSTRACT

Sasol Technology and Linde AG have developed an innovative azeotropic distillation process. A light-boiling binary entrainer (ethanol and water) is used to recover hydrocarbons from a process stream that also contains oxygenated species. Conventional distillation cannot be used because of low-boiling azeotropes that exist between the hydrocarbons and oxygenates.

The use of a binary entrainer is crucial to the success of the process concept. Each entrainer component on its own could not achieve the desired separation of octene and oxygenates.

Extensive use has been made of graphical conceptualising techniques and steady state simulations to aid process development and process synthesis. A single ternary diagram does not provide sufficient insight into the system. Hence, a paper pyramid model was constructed, by joining ternary diagrams to better visualise vapour-liquid equilibrium relationships of the four key components in this system.

Keywords: heterogeneous azeotropic distillation, quaternary systems, graphical conceptualising techniques, steady state simulations; recovery of olefins from oxygenates

BACKGROUND

Sasol Olefins and Surfactants business unit is responsible for the commercial production of co-monomer grade 1-octene product, which is recovered from the Sasol refinery streams. The first 1-octene train was commissioned in 1999 and has a capacity of 50 000 tpa. There is sufficient 1-octene available for a second octene train to be built.

The first octene train makes use of a potassium carbonate wash to remove acids from a C₆-C₁₀ hydrocarbon feed. The spent carbonate solution is regenerated in a closed loop process, which involves the incineration of the potassium organic salts formed in the wash unit. The acid-free hydrocarbon feed then undergoes pre-fractionation to remove lights and heavies and is then referred to as a C8-Broadcut. The next processing step is oxygenate removal which uses extractive distillation with NMP to remove oxygenates such as ketones and aldehydes from the C8-Broadcut.

Acid Removal and Oxygenate Removal thus occur in two separate processing steps.

Two technology options were proposed for the second 1-octene train. The first is to reproduce the existing production train. The second is to make use of a novel azeotropic distillation process to separate the hydrocarbon portion of the C8 Broadcut from the acids and other oxygenates.

This azeotropic distillation process was initially proposed and piloted with acetonitrile and water as the binary solvent. Following the suggestions of Linde-VA, more environmentally friendly solvents were investigated. The binary solvent was then changed to ethanol and water.

This process using acetonitrile and water, had been proposed for the first octene train, but due to schedule constraints, the required changes to the octene Train 1 process could not be implemented. It was realized at the start of conceptual development for the second 1-octene train, that the azeotropic distillation process with ethanol and water may well hold several operational and cost advantages over the existing Train 1 process.

These advantages include the fact that this novel technology combines the, previously separate, Acid Removal and Oxygenate Removal processing steps. The complexity of the process is significantly reduced as the tagged equipment count is reduced from 25 to 12 for this portion of the process. This provided the incentive to investigate the proposed process further, as well as embark on piloting exercises in order to prove the process concept.

PROCESS DESCRIPTION

Sasol Technology (Pty) Ltd and Linde AG have developed an azeotropic distillation process using a binary solvent to recover the hydrocarbon portion of a C8-Broadcut containing hydrocarbons, acids and other oxygenates. The hydrocarbons in the C8-Broadcut cannot be separated from the acids and other oxygenates by simple distillation due to low-boiling azeotropes that exist between the hydrocarbons and the oxygenated species.

The PFD in figure 1 indicates that the C8 Broadcut feed, containing acid and other oxygenate impurities, is fed to the azeotropic column at an intermediate feed point. The azeotropic column reflux is a recycle stream that contains a mixture of water, hydrocarbons and ethanol. The hydrocarbons in the feed form light boiling azeotropes with water and ethanol. These hydrocarbon-ethanol-water

heterogeneous azeotropes can then be recovered to the overheads. The oxygenates, including acids are recovered to the bottoms of this column.

The overhead product of the azeotropic column is condensed and sub-cooled along with the overheads from the stripper column. This combined stream is then routed to a phase separator where a light hydrocarbon-rich phase is separated from a heavier solvent-rich phase. The heavy phase which consists mainly of ethanol and water, and also some hydrocarbon species, is routed to the azeotropic column as entrainer. The light phase is mainly hydrocarbon material with some ethanol, and very little water.

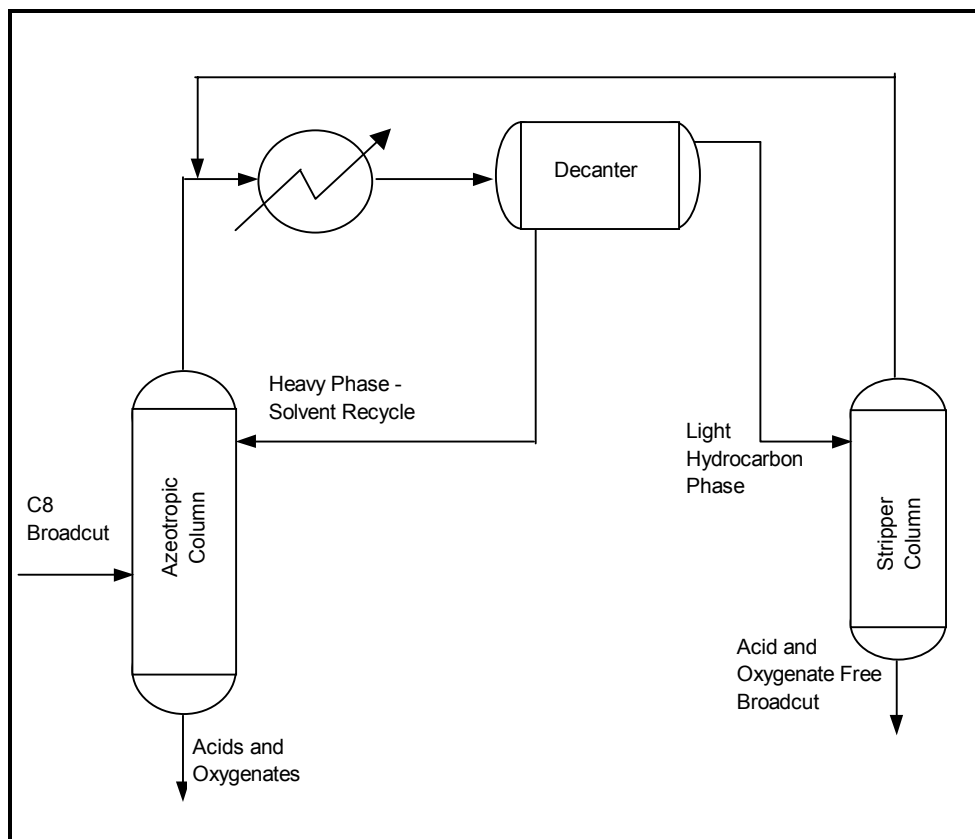


Figure 1. Process Flow Diagram of the Azeotropic Distillation Process

The light phase is fed to a stripper column where the acid and oxygenate free hydrocarbons are recovered to the bottoms. The overhead vapour product from this column is the solvent-hydrocarbon azeotrope, which is returned to the combined overheads condenser.

NOVEL TECHNOLOGY

Old and New Process Comparison

Acids are currently removed by liquid-contacting the feed using a carbonate solution, resulting in the formation of organic potassium carbonate salts. This process requires an incinerator for regeneration of the carbonate salts. Next, the remaining non acid oxygenates are removed using extractive distillation with a polar solvent.

Extractive distillation cannot be used to remove acids as the acids cannot be distilled from the polar solvent.

The azeotropic distillation process removes both acids and other oxygenates in one processing step. This process actually removes the hydrocarbons from the oxygenated components, and not the other way around. In theory, this process is not the most energy efficient for the desired separation, as the product has to be recovered to distillate, and is a greater proportion of the feed. However, in comparison to other options, it is economically very favourable. Tagged equipment count is reduced, and an incinerator is not required, which serves to reduce operating costs.

The Role of the Binary Entrainer

This is an azeotropic distillation process using a binary entrainer. Use of a binary solvent is key to the success of the process.

It is the use of ethanol that allows the hydrocarbons to be recovered from the oxygenated species. Ethanol forms low-boiling azeotropes with the hydrocarbons, but does not azeotrope with oxygenates. Use of water alone would not facilitate recovery of the hydrocarbons, since water also azeotropes with oxygenated species.

Addition of water to the system has a number of important process consequences:

Firstly, addition of water results in the formation of a minimum boiling azeotrope between the hydrocarbons, water and ethanol. It is this water-containing azeotrope which is used to recover the larger portion of the hydrocarbons. This azeotrope is heterogeneous, thereby enabling the solvent to be recovered in a phase separator (instead of requiring an energy intensive separation process).

Secondly, the water/1-octene/ethanol azeotrope is richer than the binary ethanol/1-octene azeotrope in octene content. The same is true for other hydrocarbons in the feed. This means that addition of water lowers the solvent:feed ratios required to recover hydrocarbons from oxygenates. The azeotropic column energy requirements will also be lower. The higher the water content in the solvent, the lower the solvent:feed ratio required, and also the better the phase separation obtained.

There is however an upper limit to the amount of water that should be present in the solvent. This stems from the fact that water, unlike ethanol, can form azeotropes with oxygenates. Oxygenate-water azeotrope formation is undesirable as oxygenates are to be removed in this processing step. These oxygenate-water azeotropes are lighter boiling and more difficult to distill to the bottoms than the pure oxygenates. The solvent which is recycled to the azeotropic column should always keep the system 'water lean' of the ternary azeotrope, in order not to cause acids and oxygenates to be recovered to the distillate.

In essence there should be sufficient water in the entrainer to ensure that the major proportion of the hydrocarbons are recovered with the water-containing azeotrope thus ensuring a more energy efficient process. The remaining portion of hydrocarbon material will then be recovered with the ethanol-hydrocarbon azeotropes. At the

same time, by ensuring the system is sufficiently water-lean, the availability of water to form light boiling azeotropes with the oxygenated species is limited.

PROCESS DEVELOPMENT

In the initial phases of process development, solvent screening was required in order to identify candidate solvents for the process. Solvents were first screened according to the nature of the azeotropes formed (pure component properties, azeotropic compositions, heterogeneous or homogeneous azeotropes). From this emerged two strong candidates with similar advantages as described above. The ethanol-water option was chosen due to the fact that it was the more environmentally friendly solvent.

Next, process development entailed that the process concept be synthesized, and then proven by piloting. Process synthesis was conducted by means of steady state simulation. The steady state simulation was then validated by extensive piloting.

A steady state model of the process was developed on a simulation package using a sequential modular solution algorithm. Due to the non-ideality between components in the liquid phase, the best choice for the VLE calculations was considered to be a liquid activity coefficient model. The UNIFAC-Dortmund liquid activity property method was used for simulations of the azeotropic and stripper columns. The method used is a customised version of the UNIFAC-Dortmund method, which is available exclusively to UNIFAC consortium members; of which Sasol is a member.

To simulate the phase separation in the decanter, the UNIQUAC liquid activity model was used. For this model, binary interaction parameters were regressed for the key component interactions based upon measured binary and ternary liquid-liquid equilibrium data.

MULTIPLE STEADY STATE SOLUTIONS

When the process was first piloted, two distinctly different operating modes were observed for the azeotropic distillation column. These two modes were dubbed the hot profile and cold profile modes, due to the characteristic temperature profiles that occur in the azeotropic column. These temperature profiles are associated with significantly different and unique azeotropic column composition profiles and different overhead product stream compositions (i.e. a different component mass balance exists for each mode).

These two solutions are obtained using the same or very similar simulation specifications.

During a recent piloting exercise a third operating mode was discovered. This mode is characterised by an intermediate temperature profile. It was originally dubbed a water-rich mode due to the fact that there was water and very little ethanol in the bottoms product of the azeotropic column. It was thought that water was present in excess of the hydrocarbon-water-ethanol azeotropic requirements. However, this is

not necessarily the case. This mode is definitely much closer to the point where there is sufficient water to satisfy the hydrocarbon-water-ethanol azeotrope. This mode is also associated with a unique azeotropic column composition profile as well as a unique overhead composition.

INCORPORATING GRAPHICAL CONCEPTUALISING TECHNIQUES

The 1-octene azeotropic distillation process uses a mixed solvent (e.g. ethanol and water) to separate hydrocarbons from oxygenates. The key hydrocarbon in this process is the 1-octene and the key oxygenate is considered to be 2-hexanone. Hence there are four key components in this system (i.e. a quaternary system).

Initially an attempt was made to optimise the steady state simulation of the azeotropic distillation process by performing sensitivity studies on different simulation model input as well as by analysing the ternary diagram for 1-octene, ethanol and 2-hexanone (as in Fig 2). It was soon realised that only a limited understanding of the process is obtained using this approach.

To further understand the system it was realised that a graphical method should be used that included all four key components. In the case of 4 components, graphical visualisation occurs in 3 spatial dimensions, which is difficult to portray in the 2 dimensions available on computer screens and paper.

Construction of Pyramid Model

To aid with the conceptual understanding of the quaternary azeotropic distillation process, a paper pyramid model of the quaternary system vapour liquid equilibrium was constructed. The pyramid was made by separately constructing VLE ternary diagrams for each of the four possible ternaries. The edges of the four ternary diagrams were then joined together to form a paper pyramid.

To aid discussion on the quaternary system, an example of one of the VLE ternary diagrams used to construct the pyramid is included, namely that for the ethanol/1-octene/2-hexanone system at 140 kPa abs. See figure 2 for further discussion.

There are a number of singular points on this diagram. These points are either azeotropes or pure components. Residue curve can be constructed and these lines will pass from the lowest boiling point (unstable node) to the highest boiling point (stable node). The remaining points are saddles where the residue curves approach and then move away again without touching.

The system also has a simple distillation boundary, which cannot be crossed using conventional distillation. This boundary needs to be crossed in order to recover 2-hexanone as a bottoms product without solvent or octene loss in the azeotropic column. This distillation boundary is crossed by means of addition of sufficient solvent [1,2].

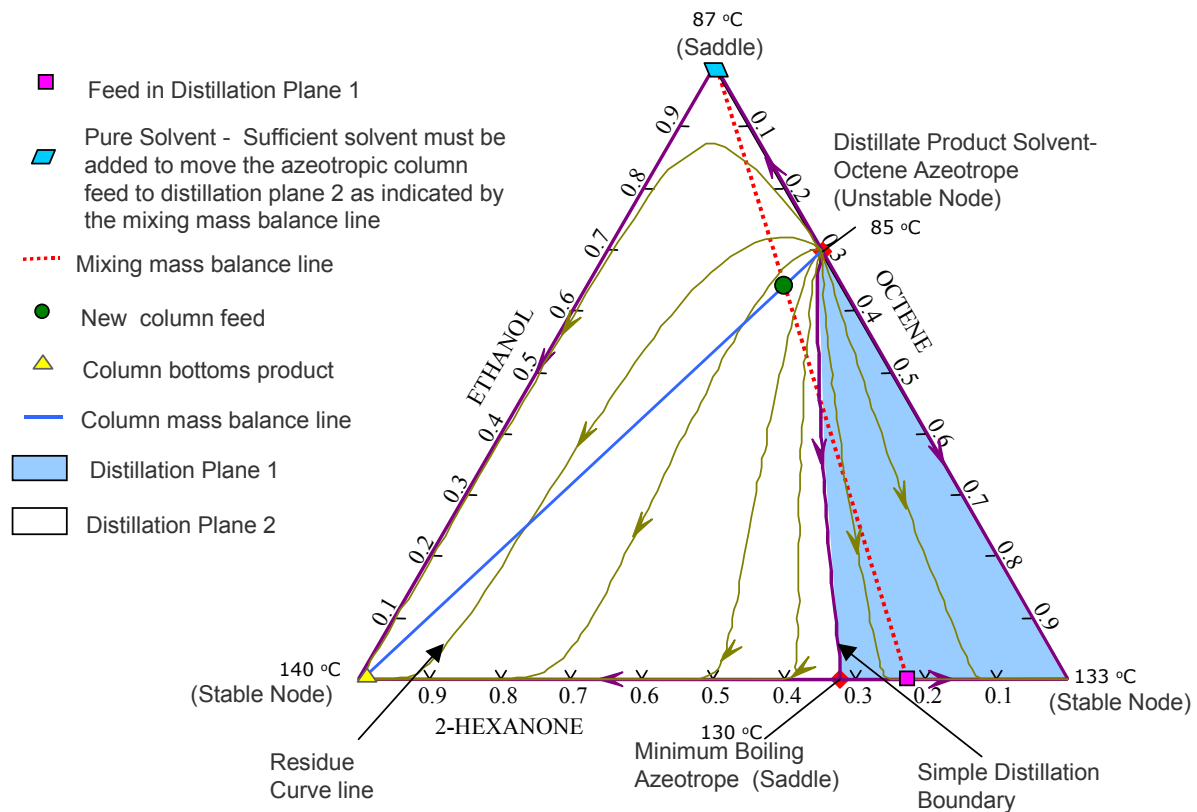


Figure 2. Ternary Map of Ethanol-Octene-Hexanone at 140 (kPa abs)

Distillation column products need to be on the same residue curve line to be obtainable. They also need to lie on the same mass balance line as the feed. The column mass balance line will thus start and terminate on the residue curve line, and pass through the feed composition [1,2].

Changes in the column mass and energy balance, or feed composition can change the residue curve on which the column products are found.

Each residue curve is associated with a unique temperature and composition profile in a distillation column. This means that the various profiles obtained in the azeotropic column are related to the product mass flow rates and compositions obtained via the residue curve and mass balance lines on which these products are found.

Description of Quaternary VLE Pyramid Model

Presented in figure 3 is a schematic diagram showing the four ternary VLE pyramid faces for ethanol/water/1-octene/2-hexanone at 140 kPa abs. Each apex of each of the four triangles represents a 100% pure component as labelled. For each of the four ternary diagrams, residue and boundaries are shown.

In the inside of the pyramid, three different volumes can be identified, which are separated by distillation boundary surfaces. The three distillation volumes represent three spaces that cannot be crossed using conventional distillation under total reflux conditions. Within each of the volumes are the singular points and residue as found for a 3-component system.

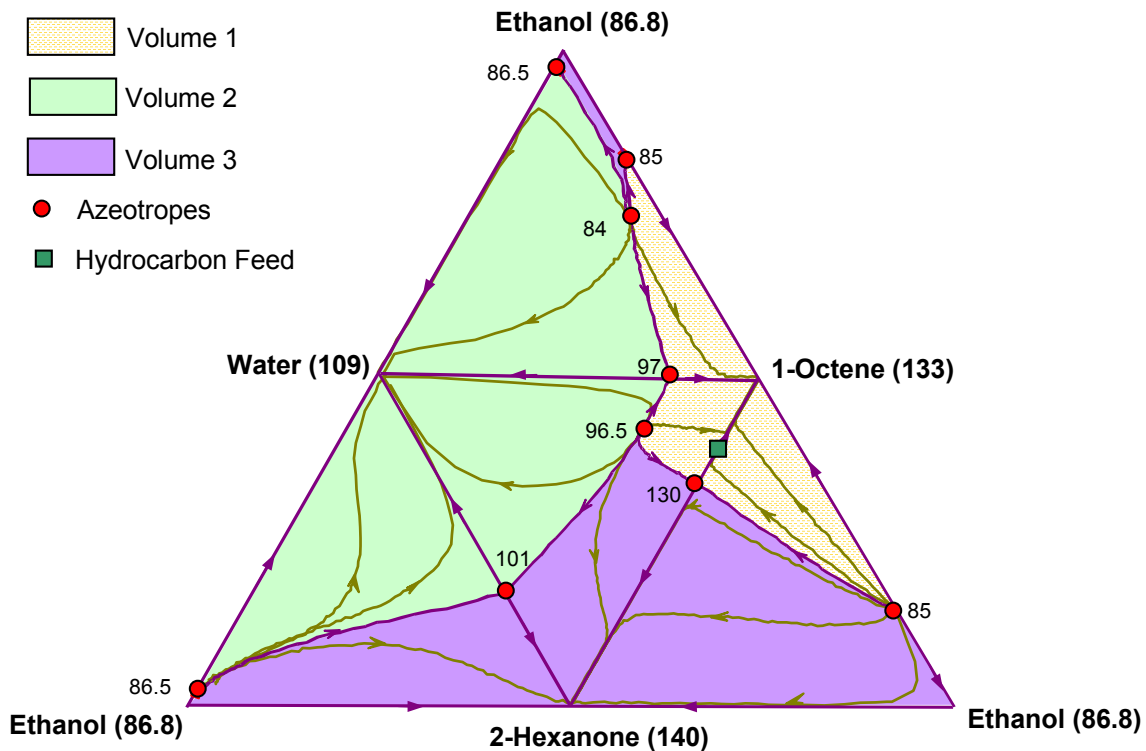


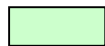
Figure 3. Pyramid Model Showing Quaternary VLE for Ethanol/Water/1-Octene/2-Hexanone with boiling points at 140 kPa (abs) in degrees Celsius.

- **Distillation Volume 1**



The first distillation volume is the smallest of the three volumes. Within this volume is the feed that is located along the 1-octene/2-hexanone axis (i.e. no ethanol/water in the feed). Within this volume, residue curves move from the ethanol/water/1-octene ternary azeotrope (boiling point 84 °C) to the stable node that is 1-octene (BP = 133 °C).

- **Distillation Volume 2**



This volume is the largest of the three volumes. Residue curves move from the ethanol/water/1-octene ternary azeotrope through to water (BP = 109 °C).

- **Distillation Volume 3**



In this volume, residue curves move from the ethanol/water/1-octene ternary azeotrope through to 2-hexanone (BP = 140 °C).

The ethanol/water/1-octene ternary azeotrope is part of volume 3 on the above diagram. It is known (from the experimental and simulation results) that this ternary azeotrope can be obtained as distillate in the azeotropic column, with a bottoms product of 2-hexanone. It is the common point between all three distillation volumes discussed above.

Description of Quaternary Azeotropic Distillation Process

In the azeotropic distillation process, the feed that is in volume 1, must be separated into a hydrocarbon product stream containing 1-octene (volume 1), and an oxygenate waste stream containing 2-hexanone (volume 3). To fractionate the feed into the two product streams, distillation boundary surfaces must be crossed. This is achieved by mixing the feed together with a mixed ethanol/water solvent.

In Figure 4., the relevant phase separation boundary at 40 °C governing the ethanol/water/1-octene system has been included on that ternary diagram to aid discussion of the process.

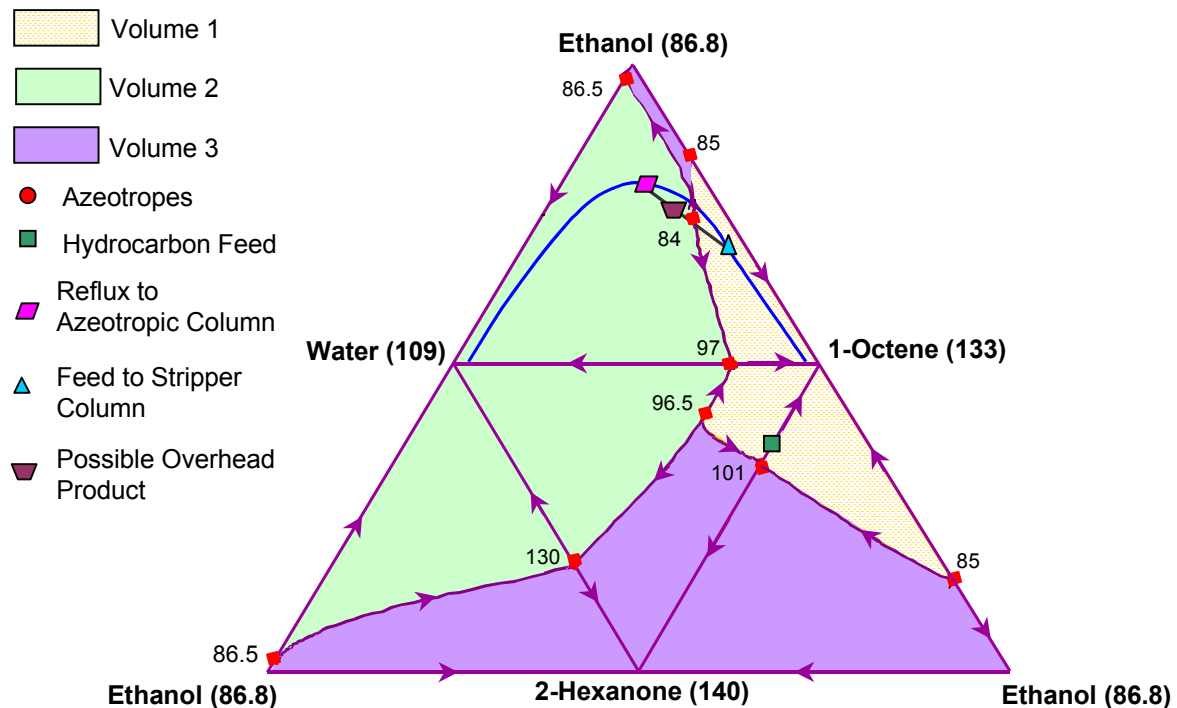


Figure 4. Pyramid Model Showing Quaternary VLE for Ethanol/Water/1-Octene/2-Hexanone at 140 kPa (abs) and LLE for the Ethanol/Water/1-Octene at 40 °C with temperatures in degrees Celsius

In the azeotropic distillation column, sufficient solvent must be added (reflux) so that the mixed feed point lies within volume 3. With the total feed inside volume 3, the azeotropic column separates 2-hexanone (bottoms) from the ethanol/water/1-octene ternary azeotrope (overheads). The azeotropic column overheads, which will be at a point close to the ternary azeotrope, is condensed upon which the condensate forms two liquid phases. The light phase (rich in 1-octene) will lie within volume 1 and the heavy phase (rich in solvent) will lie in volume 2. The heavy phase, in volume 2, is refluxed back to the azeotropic column, and serves to ensure that the total feed to this column lies in volume 3.

The light phase is refluxed to the stripper column. In the stripper column, the reflux feed is fractionated to obtain 1-octene in the bottoms and the ethanol/water/1-octene

ternary azeotrope in the overheads. The stripper overheads is then combined with the azeotropic column overheads.

Other Insights gained from the Quaternary Representation

Being able to visualize part of the interaction between all four components in the system lends itself to other important insights.

The first concerns the role of the solvent. It can now be seen that a solvent containing too much water would move the combined azeotropic column feed to volume 2 instead of volume 3. In this volume, water is the stable node, and not 2-hexanone. There is thus an upper limit to the water content in the azeotropic column reflux.

Ethanol is the component in the azeotropic column reflux that ensures preferential overhead recovery of 1-octene in the feed from 2-hexanone, by the formation of a lower boiling azeotrope. It can be seen from the liquid-liquid equilibrium (LLE) that too much ethanol in the system would result in loss of phase separation.

The C8 Broadcut is a 'given' input to this system, and is a feed lying in volume 1. If however, the feed were richer in oxygenated species, it would already lie in volume 2. The azeotropic column would then require a lower solvent:feed ratio. Recycling the oxygenate impurity would be of limited economic benefit for this system which has a fixed throughput, and would only serve to increase equipment sizes.

Comparison of Operating Modes – Simulation Results for the Azeotropic Column

In order to better understand this azeotropic distillation process, and attempt to gain an understanding of the causes of each mode, simulations were completed using only the 4 key components that define the process. The 4-components system is a simplification of the real system.

Utilising the quaternary system facilitates understanding of the process in terms of the mass feed rates required to obtain the desired products from the process. The next step is simulation and piloting. As mentioned in previously, this process appears to have multiple solutions or operation modes.

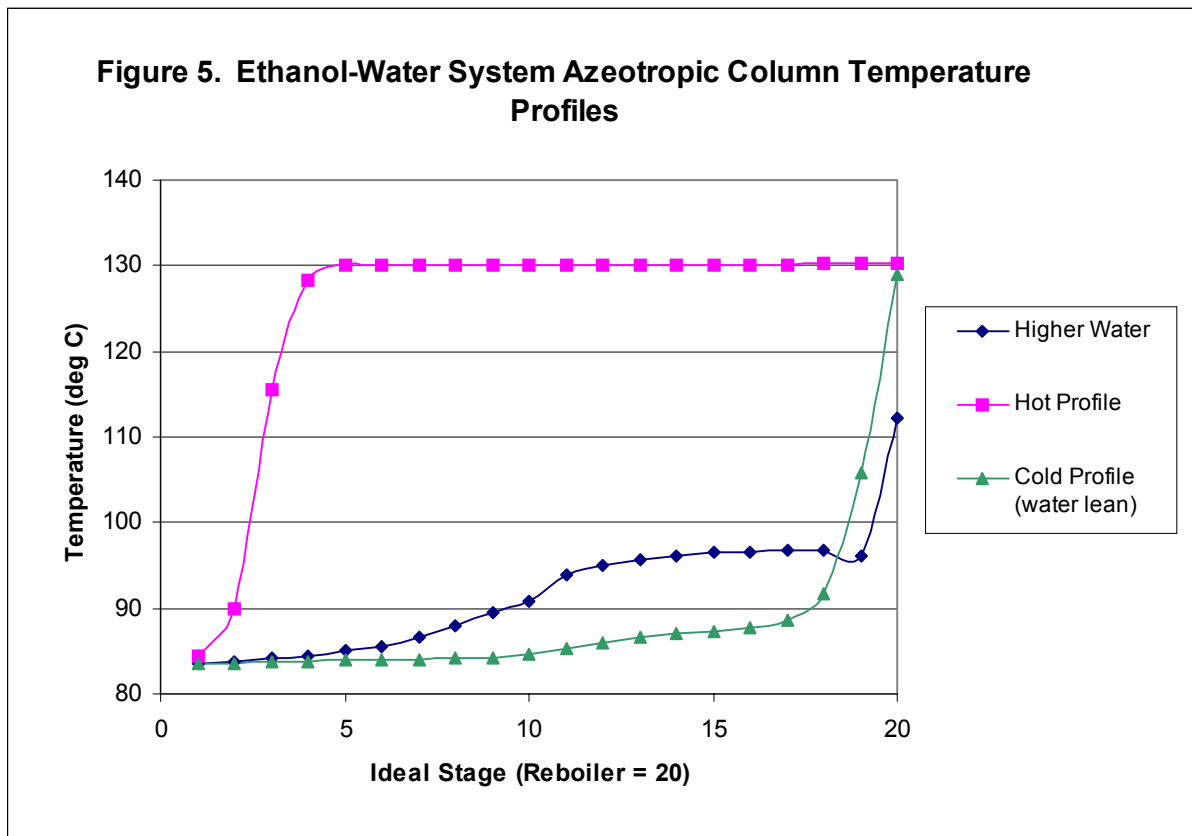
For the simulation results shown, open loop simulations with respect to the solvent composition were used (i.e. reflux composition is a simulation input specification). This is because for two of the solutions obtained, a converging solution is not possible due to component build up. (This is explained in more detail in the following section).

In, addition, the liquid-liquid equilibrium (LLE) describing the phase separator was predicted using the UNIFAC-DMD property method.

In table 1 it can be seen that the hot and cold profile solutions occur at exactly the same simulation input. The higher water mode is found at higher solvent:feed ratios. Figure 5 depicts the drastic differences in temperature profile associated with each of these simulation solutions.

Table 1. Simulation Input and Results for three solutions describing the Azeotropic Column

| Simulation Input | Cold Profile (Water Lean) | Hot Profile | Higher Water Profile |
|------------------------|------------------------------|-------------|-------------------------|
| Feed Flow (kg/h) | 100 | 100 | 100 |
| Solvent Flow (kg/h) | 326 | 326 | 357 |
| Bottoms Flow (kg/h) | 20.5 | 20.5 | 20.5 |
| Ohd Pressure (kPa abs) | 140 | 140 | 140 |
| Results | | | |
| Ohd Temp (oC) | 83 | 85 | 84 |
| Feed Stage Temp (oC) | 85 | 130 | 91 |
| Reboiler Duty (kW) | 94.7 | 95.2 | 102.8 |
| Ethanol Bttms (wt%) | 2.34 | 0.00 | 0.11 |
| 1-Octene Bttms (wt%) | 0.01 | 57.11 | 0.00 |
| Water Bttms (wt%) | 0.09 | 0.00 | 2.38 |
| 1-Octene recovered (%) | 100.00 | 92.57 | 100.00 |
| 2-Hexanone removed (%) | 100.00 | 43.96 | 99.52 |

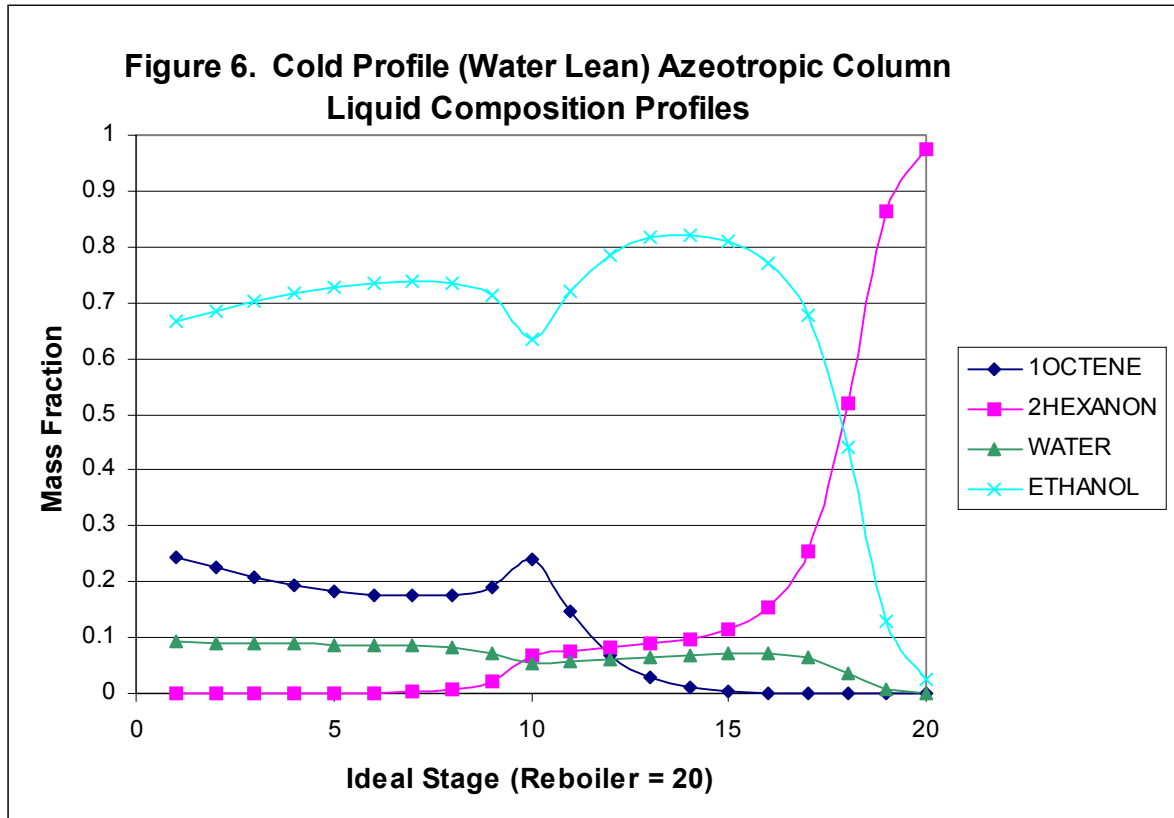


Cold Profile (Water Lean)

This solution is characterised by low temperatures (a few degrees warmer than the overheads) at and above the hydrocarbon feed point. In figure 6, it can be seen that ethanol and water are present in the composition profiles in high concentrations from the top stage to well below the hydrocarbon feed point. This solution shows excellent octene recovery as well as excellent oxygenate removal.

Simulations of this option tend to predict ethanol and water losses to the bottoms of the azeotropic column. Simulations done by Linde-AG during development of the process concept show that it is possible to obtain a cold profile without ethanol losses to the bottoms of the Azeotropic column. It has also been shown in previous piloting attempts that operation in this mode is possible without losses of solvent to the bottoms.

In the event that solvent does appear in the bottoms stream, the effects on the downstream processing units need to be quantified. Ethanol is not likely to be a serious problem.



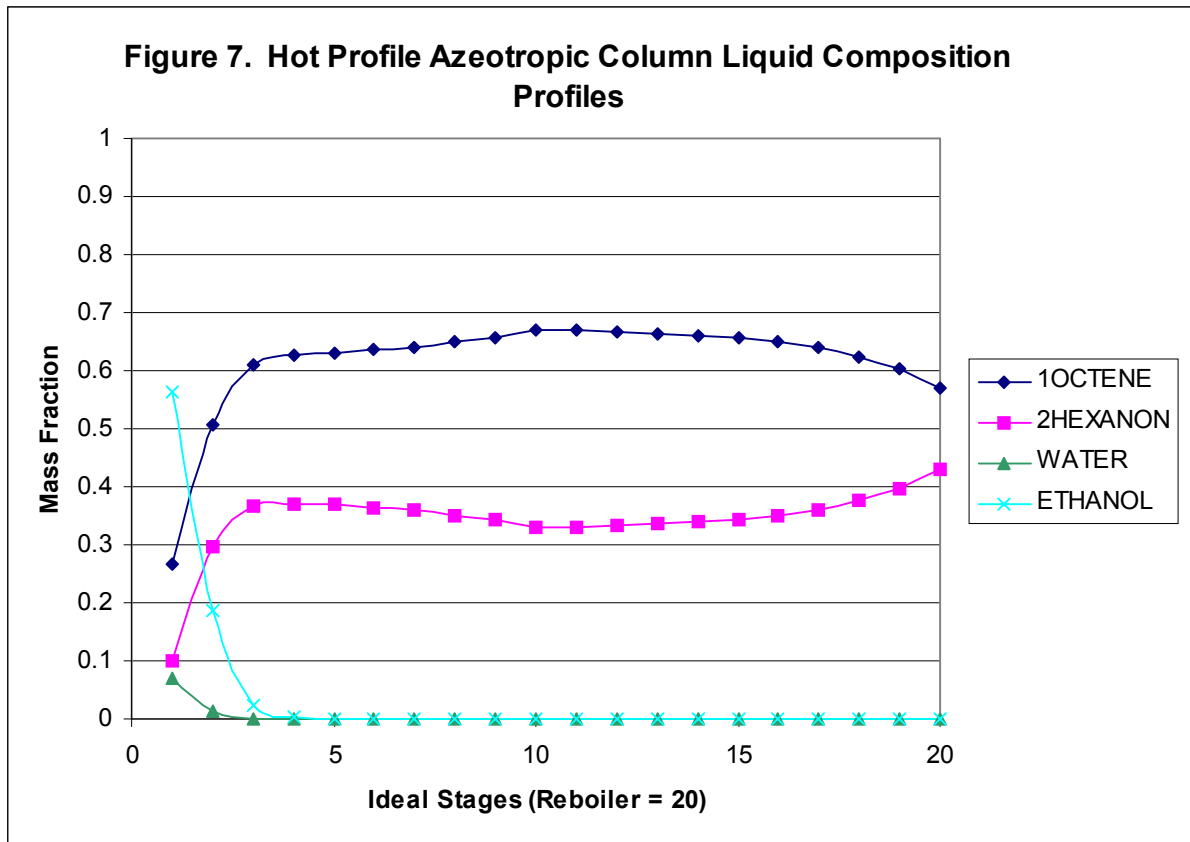
Hot Profile

This solution is characterised by temperatures well above the distillate temperature at and above the hydrocarbon feed point. The composition profiles in figure 7, show that solvent and water are quickly depleted and are at negligible concentrations at the hydrocarbon feed point. Because of the rapid depletion of solvent, there is not much separation between 1-octene and 2-hexanone.

This solution is characterised by octene loss, however hot solutions can also be obtained at high (95%+) octene recovery. The other significant characteristic of this profile is that oxygenate removal is not optimal. This profile can be achieved with varying proportions of 2-hexanone removal, from poor (60%) to good (ca 95%).

The implications of improper oxygenate removal are important. Light oxygenates (not included in this simulation) will build-up in the solvent loop. Heavier Oxygenate species such as 2-hexanone will build up to an equilibrium level in the solvent loop,

and are not expected to cause loss of phase separation in the phase separator. All oxygenated species will also be present in the light phase feed to the stripper and so contaminate the 1-octene product from the stripper.



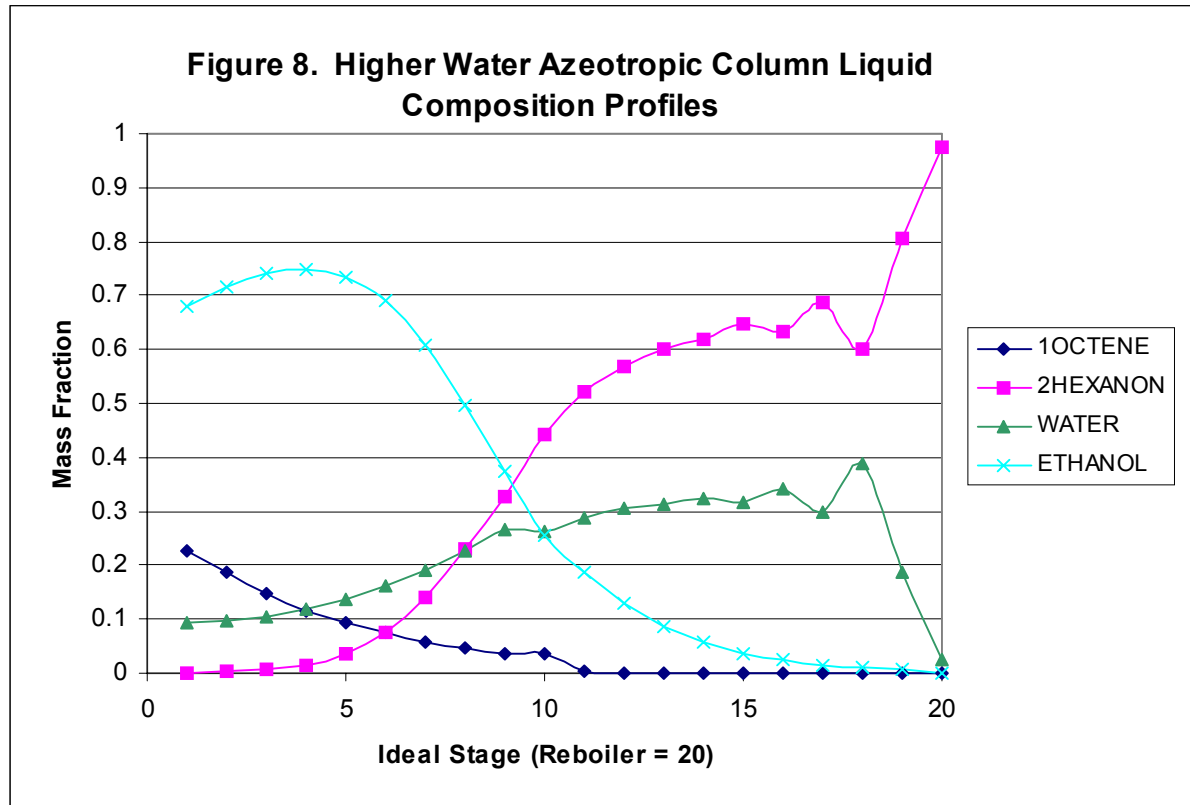
Higher Water Cold Profile

This solution is characterized by an intermediate temperature profile, but one which is more similar to the cold profile solution. Looking at the composition profiles in figure 8, it can be seen that while ethanol is present below the feed point, it is not present in as high concentrations as for the water lean cold profile solution. What is significant about this profile is that water is present below the feed stage in far higher concentrations than for the water lean profile. In fact water penetrates far deeper than ethanol. Furthermore, hexanone is also present in the top section of the column.

This solution is simulated to have water, but very little ethanol in the azeotropic column bottoms product. The simulations also predict that there will be phase separation in the azeotropic column below the feed point.

Experimental data has been gathered which describes operation of the azeotropic column for a water-rich solvent recycle. It was seen that the column maintained a temperature profile similar to the simulations, and that the temperature does increase with increasing oxygenate presence in the liquid on the trays above the feed point. The occurrence of two liquid phases was not reported. The bottoms product contained water but also no ethanol.

This mode was simulated by increasing the solvent:feed ratio. It can also occur because the solvent is rich in water, which means that a lower solvent:feed ratio can be used to recover the hydrocarbons than for the water-lean case (as happened during the piloting). This is a more favourable case for plant capital and reboiler operation economics than these open-loop simulation results would indicate.



Process Implications of each operating mode

The hot profile is obviously not a desirable mode of operation, as it does not achieve the objective of acid and other oxygenate removal.

The higher water cold profile simulations seems to dispel fears that too much water will cause oxygenate carryover as it has been seen in piloting that it is possible to keep oxygenates out of the distillate. This was achieved by operating the azeotropic column so that excess water left the column via the bottoms product. To remain in this higher water operation mode, water would have to be made-up as a result of the water-losses to the azeotropic column bottoms. This option should only be considered if there are problems with the phase separation as this is the option that will ensure maximum water content in the phase separator.

The water lean cold profile is the most desirable operational mode, as there is no risk of oxygenate carryover when there is high product recovery. Experimental data shows that there is no solvent loss either.

FURTHER DISCUSSION ON THE QUATERNARY SYSTEM

What is also of interest is that all three these simulation solutions lie within the same distillation volume. Closer examination of the quaternary system is required to

understand the causes of the different solutions. At this stage it is known that the solutions lie in different residue curves, due to the different component mass balances that govern the separations.

With very similar overall column mass balances (bottoms:feed ratios), different azeotropic column results are obtained. In other words, small movements in the feed point (as plotted in the ternary diagram for illustration) or small changes in the column mass balance line, can cause that the column mass balance line, begins and ends on totally different residue curves in the quaternary diagram.

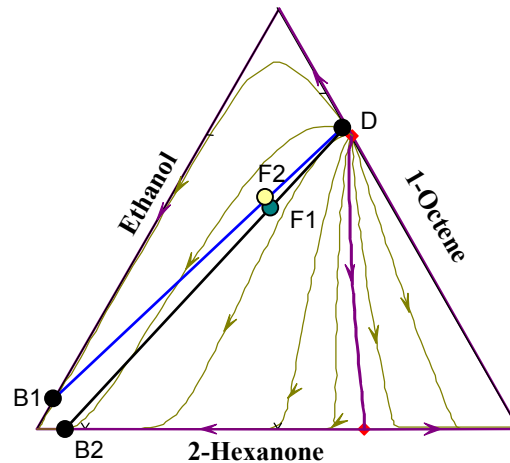


Figure 9. Different distillation column products obtained on different residue curve lines due to small changes in the feed composition

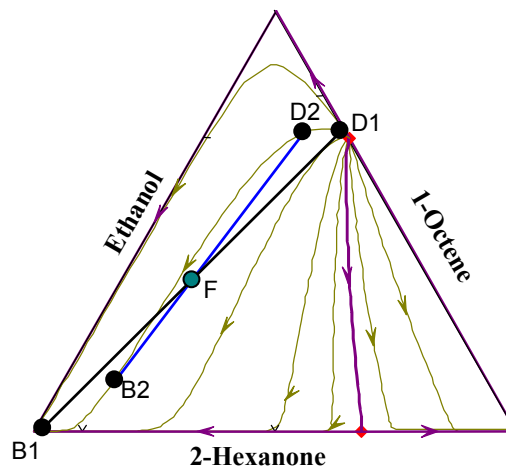


Figure 10. Different distillation products obtained on the same residue curve due to changes in distillation column mass balance ($D:F$ ratios)

Figure 9 and 10 depict two scenarios in which the combination of column mass balance, and feed composition determine the residue curves on which the products lie, and thus the column composition and temperature profiles. This is done for the ethanol-octene-hexanone system, but the same reasoning can be applied to any 3-component system, or multi-component systems.

In the case of the cold and hot profile, it would appear that for the same feedpoint, and mass balance line, there exist more than one residue curve on which the column

mass balance line can start and terminate. This is because the mass balance line can be orientated differently in the 3-dimensional space. The residue curve on which the mass balance line starts and terminates, is determined by the operating conditions of the azeotropic column.

This sensitivity in simulation solutions could be because the total column feed, can only be moved into a limited region within volume 3. The column feed is in fact in a volumetric space near the ternary azeotrope where all the residue curves are converging. This would mean that only small column feed composition changes are needed to move to a different residue curve (for the same or different mass balance line). This change in azeotropic column feed composition, would occur as a result of a solvent flow rate increase or decrease, or as a result of a solvent composition change.

Table 2. Total Azeotropic Column Feed Composition

| Component | Cold Profile (Water Lean) | Hot Profile | Higher Water Profile |
|-----------|------------------------------|-------------|-------------------------|
| | wt% | wt% | wt% |
| 1OCTENE | 37.0 | 37.0 | 36.3 |
| 2HEXANON | 4.7 | 4.7 | 4.4 |
| WATER | 7.5 | 7.5 | 7.6 |
| ETHANOL | 50.8 | 50.8 | 51.7 |

The above would imply that careful thought around the control philosophy will be required to ensure that the optimal azeotropic column profile is maintained.

CONCLUSIONS

Steady state simulations have been used with success as an aid for process development. They were used in the early stages of process synthesis, as well as to set up the guidelines for piloting experiments. Simulations were then verified with piloting data. They were also used as an aid to understanding piloting data. Thereafter they were used to optimise the operating parameters governing the process at steady state.

The understanding of this azeotropic distillation process was greatly enhanced by the use of graphical techniques, and especially by attempting to visualise the 3-dimensional system. The insight gained from the use of these graphical techniques has explained some of the physical phenomena observed in piloting. It has also resulted in a greater understanding of the reasons for upper limits for the water and ethanol content in the solvent.

Processes using azeotropic distillation for separation of non-ideal mixtures are complex to design, even more so when the process uses a binary entrainer. Further development of modeling tools, allowing a 3D visualization of these problems is required. This ability to visualize the separation, in combination with research into attainable regions would aid understanding of the complexities of the system and make it possible to reach a more optimal design.

While piloting results have not been discussed, it should be noted that piloting experiments have also played a significant role in development of this process. Design of an adequate control system will be required in the next stages of process development.

Neither the steady state simulations, nor the graphical techniques for understanding this azeotropic distillation process replace the need for piloting experiments. Both techniques enhance the understanding gained from piloting.

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REFERENCES

The following persons have also written reports that would have been used as references, but are not, as these are Sasol Technology (Pty) Ltd and Linde AG proprietary documents.

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In addition to the proprietary references, the following are excellent summary references:

1. Widagdo S., Sieder W. D., 'Azeotropic Distillation', AIChE Journal, January 1996 Vol 42, No.1, pg 96-130

2. D. Glasser, D. Hildebrandt, B. Hausberger, Course notes from the University of the Witwatersrand, (South Africa), Chemical Engineering Department, Distillation Synthesis: Residue Curve Maps as a Tool for Multi-Component Distillation (2001)