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Analysis of the recovery of aqueous solvents from the pharmaceutical industry: isopropanol and ethyl acetate

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Change is the end result of all true learning.

Leo Buscaglia

En primer lloc agrair als meus pares el suport i ajuda incondicional que m'han donat durant aquests anys de carrera, i per tot l'esforç realitzat a l'hora d'ajudar-me a formar-me i ensenyar-me a intentar ser la millor persona possible. També dir que estic orgullós d'haver pogut completar el que un dia el meu pare no va poder.

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Agrair especialment als meus amics i companys de tota la vida i els que he conegut al llarg de la carrera, que han sigut una part indispensable i fonamental a l'hora de poder superar aquesta etapa i per haver compartit tants moments inoblidables.

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SUMMARY

It is crucial to work with as few compounds as possible in the pharmaceutical industry when recovering different products, as any compound added to the mixture leaves traces. In the specific case of this study, the recovery of isopropyl alcohol, ethyl acetate and water from the production of a commercial antibiotic called ampicillin is studied.

In the literature, the use of an extracting agent is proposed to facilitate their recovery due to ethyl acetate, and isopropyl alcohol form a complex non – ideal mixture that makes their separation difficult. However, considering that ethyl acetate and water have a zone of immiscibility allows the separation without requiring extractive agents.

In this study, different feasible alternatives are proposed, which have been obtained employing the analysis of the residue curve maps and they are compared through the DSE (Distillation Sequence Efficiency) method. The DSE allows the calculation of the efficiency of rectification columns quickly using ∞/∞ analysis.

The results show that out of the five proposed alternative, three can be implemented, and two of them use a smaller number of rectification columns than the one proposed in the article. In terms of efficiency, one alternative has a similar DSE to that of the literature but without extracting agents, which is an improvement.

Keywords: Distillation, DSE, Residue curve map, Aspen Plus®, Azeotropes

RESUM

En la indústria farmacèutica, és crucial treballar amb el menor nombre de compostos a l'hora de recuperar diferents productes, ja que qualsevol compost afegit a la mescla deixa traces. En el cas concret d'aquest estudi, s'estudia la recuperació de l'isopropanol, l'etil acetat i de l'aigua, que s'obtenen de la producció d'un antibiòtic comercial anomenat ampicil·lina.

En la literatura es proposa fer servir un agent extractor per a facilitar la seva recuperació, pel fet que, l'etil acetat i l'isopropanol formen una mescla complexa no ideal que ho dificulta. Tot i això, en aquest no es té en compte que l'etil acetat i l'aigua presenten una zona d'immiscibilitat, la qual cosa permet la seva separació sense requerir l'adició d'agents extractors.

En aquest treball, es proposen diferents alternatives factibles que s'han obtingut per mitjà de l'anàlisi dels mapes de corbes de residu i es procedeix a comparar-les per mitjà del mètode DSE (Distillation Sequence Efficiency). El DSE permet calcular l'eficiència de columnes de rectificació de manera ràpida per mitjà de l'anàlisi ∞/∞.

Els resultats mostren que de les 5 alternatives proposades, 3 es poden implementar a la realitat i que dues d'elles utilitzen un nombre menor de columnes de rectificació que el proposat en l'article. Pel que respecta a l'eficiència, una alternativa té un DSE molt similar al de la literatura, però sense tenir agents extractors, el que representa una millora.

Paraules clau: Destil·lació, DSE, Mapa de corbes de residu, Aspen Plus®, Azeotrops

1. INTRODUCTION

There is evidence that it is crucial to work with the least quantity of compounds in the pharmaceutical industry to recover different products from having the least number of traces.

In this case, this study focuses on producing an antibiotic, ampicillin sodium, which is widely used in daily life. Ampicillin sodium is a semi-synthetic antibiotic with a broad antibacterial spectrum, low toxicity, high efficacy, and low price, favouring the market prospects [1].

However, producing ampicillin sodium causes pollution caused by the wastewater generated containing ethyl acetate (ETAC) and isopropyl alcohol (IPOH). However, if it is possible to separate these compounds and recirculate them, the pollution and production cost will be reduced[2].

Distillation is the most common unit process for separating liquid mixtures because of its advantages in operation and control. Nowadays, approximately 95% of all liquid separation is carried out by the distillation process. Despite these advantages, for systems with similar boiling points and azeotropes, the conventional distillation fails to obtain a rich phase volatility. Hence separating azeotrope systems is not possible. To carry this out, the most common techniques for separating this kind of mixtures are membrane separation techniques, pressure–swing distillation, extractive distillation or azeotropic distillation.

The main problem of separating these compounds is a ternary azeotrope system of ethyl acetate – isopropyl alcohol - water and three binary azeotropes. This makes separation difficult with high purity by conventional distillation. Therefore, it is necessary to break this azeotrope.

Previous research has established that it is possible to recover these compounds with the aid of extractive agents such as dimethyl sulfoxide (DMSO) or ethylene glycol (EG) [3]. Nevertheless, these studies do not consider that ethyl acetate and water present an immiscible region, allowing the separation without requiring any extractive agent.

This study will examine how to make this separation possible, and different processes will be proposed studying the feasibility with the residue curve maps. Then a selection will be made by calculating DSE, and finally, a rigorous simulation will be done.

1.1. AZEOTROPES

An azeotrope is a mixture of two or more liquids that maintains their composition and boiling point during distillation at a given pressure. This mixture can either have a higher or lower boiling point than either of the compounds. This occurs when a mixture is boiled to produce vapour with the same composition as the liquid.

Azeotropes may be categorized according to three parameters: number of components, miscibility, or boiling point.

- **The number of components**: An azeotrope can be formed of different numbers of compounds. It can be formed by two components, known as a binary azeotrope. If an azeotrope consists of three compounds is a ternary azeotrope. It can also have an azeotrope with more than three components.
- **Miscibility**: There are two types of azeotropes depending on their miscibility. It can be a homogeneous azeotrope if the liquids are miscible. The other option is a heterogeneous azeotrope which consists of liquids that are immiscible and form two liquid phases.
- **Boiling point**: There are two classifications: Positive or negative azeotropes. A positive azeotrope or minimum – boiling azeotrope forms when the mixture boiling point is lower than of any of its compounds. On the other hand, a negative azeotrope, or maximum – boiling azeotrope forms when the boiling point of the mixture is higher than that of any of its components.

It is possible to predict vapour–liquid equilibrium (VLE) for a non – ideal mixture based on azeotropic condition by modifying Raoult's law. Raoult's law is valid for low to moderate total pressure and when the vapour can be assumed to be an ideal gas. But by adding the activity coefficient and the saturation pressure of each component, it is possible to predict azeotropes from non – ideal mixture [4], and they know their ideal deviation by equation 1.

$$y_i \cdot P_T = \gamma_i \cdot x_i \cdot P_i^{sat} \quad (1)$$

Where y_i is the mole fraction of component I in the vapour phase, xi is the mole fraction of component "i" in the liquid phase, P_i^{sat} is the vapour pressure of pure species, and γ is the activity coefficient of component "i" in the liquid phase.

The activity coefficient is a factor that indicates the ideal deviation in a mixture. When the activity coefficient is equal to one, the mix is ideal, and equation 1 is reduced to Raoult's law.

However, in non-ideal mixtures, this factor may have positive deviations ($\gamma > 1$) or negative variations ($\gamma < 1$) from Raoult's law.

As shown in Figure 1, the positive deviation from Raoult's law presents a minimum boiling point while the negative deviation shows a maximum boiling point.

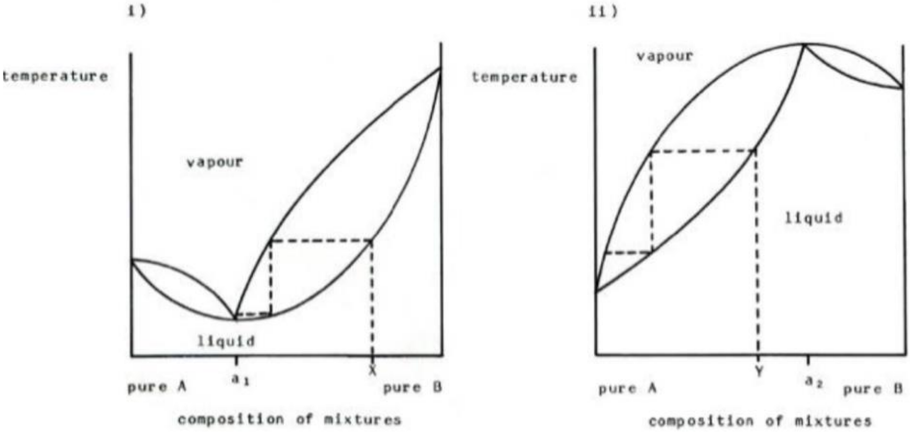


Figure 1: Boiling temperatures diagrams for non-ideal mixture i) Positive and ii) negative deviation from Raoult's law

(Phase Equilibria and colligative properties, 15/05/2021 via Chembook)

At the maximum and the minimum boiling point, the liquid phase and vapour phase equilibrium have the same composition. Moreover, in these points, the mixture boils at constant temperatures. The compounds will have the same volatility; hence the boiling liquid produces vapour with the same composition as that in the liquid phase.

1.2. ENHANCED DISTILLATION

The azeotropic distillation is crucial because the azeotrope cannot separate or is hardly done by conventional distillation. In that case, there are different methods to modify the equilibrium, allowing to obtain pure compounds. The most common are the following:

- Pressure swing distillation. A pressure swing distillation does not require any new component. It consists of separating the pressure between columns to separate a pressure-sensitive mixture with a close boiling point. Some azeotropic mixes are able to lose azeotropic behavior when the system changes its pressure.
- Extractive distillation. By adding an extracting agent, this compound is used to separate two substances with similar boiling points by changing the boiling point of one compound, making the separation easier. The extractor agent has not to form an azeotrope with the other components in the mixture. It is a suitable method for mixtures that have a low value of relative volatility.
- Heterogeneous azeotropic distillation. The advantage of this distillation is that during the distillation, the liquid phase of the mixture is completely immiscible. This causes that there can be two liquid phases on the plates, and the top vapour condensates in two liquid phases, which can be separated in a decanter.

Pressure swing distillation is particularly useful in a pressure-sensitive mixture. Nevertheless, the mix of this study is not, therefore, increasing or decreasing. The system pressure does not mean that the azeotrope will disappear.

As far the extractive distillation, as it said before, is not a suitable method, in this case, adding three compounds in the mixture could leave traces, which is a considerable inconvenience, because these compounds will be used to make medicines. This method is the most common in the literature.

Thus, this work will focus on taking advantage of the immiscibility of the ethyl acetate with water to try to break the azeotrope without the need of adding an extracting agent.

1.3. PROPOSAL FROM THE LITERATURE

Hanwen Ge et al 2020 [3] analyzed a feasible sequence to separate the ternary azeotrope mixture of ethyl acetate, isopropyl alcohol, and water. The main objective is obtaining water with small proportions of these compounds, so it can be neglected.

In figure 2, there is they propose using dimethyl sulfoxide as extractive agent to make easier the separation of these compounds:

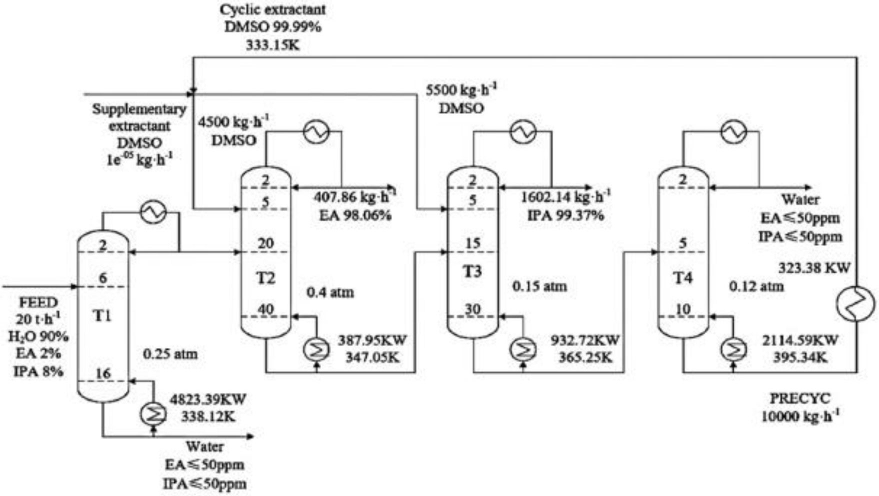


Figure 2: Proposal from the literature setup

(*extracted image from Hanwen Ge et al. 2020, Fig 8)*

As shown in figure 2, the crude feed is 20 tons/h with a mass composed of 90% water, 2% ethyl acetate and 8% isopropyl alcohol. This mixture is fed the first column, where the bottom gets pure water and is a mixture of ethyl acetate and isopropyl alcohol. This stream is provided to the second column, where the extractive agent (dimethyl sulfoxide) is added.

By the reason that DMSO has a better affinity with water and IPOH, at the top, it can obtain ETAC with 98% of purity. A mixture of IPOH, water and DMSO is collected at the bottom and fed to the third column.

The third column also has a supplementary feed of DMSO. This extractive agent has more affinity with water than IPOH. This fact allows collecting pure IPOH at the distillate. At the bottom, a binary mixture of DMSO and water feed the fourth column.

Eventually, the fourth column recirculated the DMSO obtained at the bottom, in the first column. The pure water is collected at the top.

As said previously, this work will try to improve this setup by removing the DMSO as an extractive agent. It will be verified if it is possible and if the supposed increase in economic cost that this implies is worthwhile.

1.4. PROPERTIES OF THE MIXTURE

Table 1 provides the physical properties of the compounds obtained by Aspen Plus®.

Table 1: Physical properties

Compound	Water	ETAC	IPOH	DMSO
Boiling point [K]	373.2	350.2	355.6	462.2
Critical point [K]	647.1	530.0	508.7	720.0
Specific gravity [kg/m^3]	1.00	0.89	0.79	1.10
Molar mass [g/mol]	18.01	88.11	60.06	78.13

As Table 1 shows, the three main compounds have a similar boiling point, which implies that an azeotropic distillation is required.

The different azeotropes at atmospheric pressure are presented in Table 2. The effect of pressure on the azeotropes and the detailed classification will be discussed in the methodology section.

Table 2: Azeotropes at atmospheric pressure obtained from Aspen Plus®

Temp [K]	353.33	348.51	344.76	344.61
Classification	Saddle			Unstable node
Type	Homogeneous		Heterogeneous	
Components	2		2	3
Water	0.327	-	0.327	0.321
IPOH	0.673	0.288	-	0.065
ETAC	-	0.712	0.673	0.614

1.5. MULTIPLE STEADY STATES

Multiplicities are a phenomenon that can occur when the same set of operating parameters exhibits multiple steady states. I.e. It is possible to obtain different outputs (compositions of distillate or bottom) with the same column characteristics (same feed, distillate, bottom, reflux and boil up molar flux, number of stages, feed location).

The first reported simulation about the possibility of multiple steady states for a ternary mixture with non – ideal vapour – liquid equilibrium was Magnussen et al. 1979. His study presents the possibility and the existence of three steady states for the mixture of ethanol-water – benzene. The results of his research cause a genuine interest in this phenomenon in distillation.
Multiple steady states are independent of the thermodynamic model used.

In the case of operating in the ∞/∞ model (infinite reflux and an infinite number of trays), it is possible to construct bifurcation diagrams on physical grounds to identify the multiplicities.

The prediction of the existence of multiplicities has relevant implications in the operation of real distillation columns insomuch as it is possible to obtain a completely different composition with the same operating parameters. The consequences of the multiple steady states for distillation simulation, design and operation are several [5].

These multiplicities may cause problems during the simulation due to the program can converge to one solution that is not the most attractive and possibly solved. To solve that, it is necessarily known that there are multiplicities and force the program to find the stable breach solution that the problem requires.

Mathematically, it is possible to obtain the multiplicities and it is possible to demonstrate using residue curve maps. Figure 3 is an exaggerated example of the different path that can be obtained with a given feed.

The figure below illustrates three possible paths. The first one is a →F→γ; the second one could be H→F→ε and finally c→F→δ. These three possible solutions have the same composition of the feed and mathematically may have the same distillation and a bottom molar flow rate.

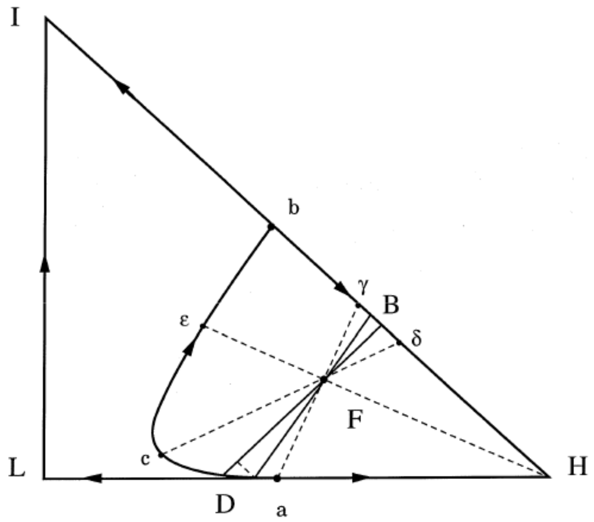


Figure 3: Multiplicities obtained at given feed

(extracted image from Bekiaris et al. 1996, Fig 2.14)

Outside of the simulation, when it is necessary to make a column that operates a mixture with these conditions, what is to be done is fill the column with the desired mix. This means that once the system is running, it tends towards it. The multiple study states are studied in depth by Bekiaris et al 1995 [6].

2. OBJECTIVES

The aim of this project focuses on the following points:

- Verify if the mixture can be separated without adding any third compound to the mix using residue curve maps.
- Study the feasibility of the alternative process proposed using the residue curve maps and carry out the material balance.
- Through the distillation sequence efficiency, critical comparison of existing and proposed methods will be made.
- Compare the alternatives from an environmental point of view.
- Compare the results and methodology of the alternative proposed with previous works (Hanwen Ge, Fanxu Fan, Guanro Su and Xiao-Hong Wang) [3].

3. METHOD

A fast to the rigorous procedure illustrated in Figure 4 is followed. The method is based solely on the thermodynamic data, which is visualized using the residue curve maps. Applying the ∞/∞ analysis, a feasible process scheme is proposed, and the corresponding mass balances are solved. A shortcut mode bases on the SEPs unit is implemented in Aspen Plus, and the corresponding distillation sequence efficiency is calculated. The procedure follows a fast to rigorous model and finally, the process schemes previously proposed are rigorously simulated.

Figure 4:Outline of the steps

3.1. SOFTWARE TOOLS

The main software used to realize this study is Aspen Plus V11® (Aspen Technology, Inc.,2021) [7]. Aspen allows to simulate processes and predict their performance by defining the thermodynamic model and the compounds to be used. Furthermore, it is possible to generate the residue curve map of the compounds in order to study the feasibility. The thermodynamic model chosen is NRTL. The parameters of NRTL (No Random Two Liquids) are adjusted to the activity coefficient obtained from data phase equilibrium empirically determined. Table 3 shows NRTL parameter values used.

Table 3: NRTL parameters obtained from Aspen Plus®

Component i	Component j	Source	T Lower [K]	T Upper [K]
Water	ETAC	APV110 LLE-ASPEN	273.150	343.55
Water	IPOH	APV110 VLE-IG	298.150	373.15
ETAC	IPOH	APV110 VLE-IG	313.150	354.15
A_{ij}	A_{ji}	B_{ij}	B_{ji}	C_{ji}
9.463	-3.720	-1705.683	1286.138	0.2
6.828	-1.312	-1483.457	426.398	0.3
2.429	-4.280	-554.632	1431.633	0.3

These parameters are from vapour liquid equilibrium data regression. The Aspen Physical Property System has various sources to determine. The source used is APV10 VLE-IG (ideal gas assumption).

Material balances are solved using Wolfram Mathematica® V.12.1(Wolfram Research, Ltd. 2021) [8] and Xcas® V.1.7 (Institut Fourier, 2021) [9]. Xcas is a free online computer algebra system that solves systems of equations, function graphs and makes interactive geometry. On the other hand, Mathematica is a computational software program used in engineering, mathematics, and computational areas. Mathematica has the same options as Xcas but is a commercial software program and can operate with more functions and do more specific operations.

Last but not least, DSE is calculated using Visual Basic Application, a macro language incorporated in Microsoft Excel® (Microsoft, 2021).

3.2. RESIDUE CURVE MAPS

Residue curve maps are a qualitative tool that allows visually how a process behaves. These represent the possible profiles of rectification column composition operating at an infinite flow rate[10]. Residue curve maps are formed by different elements, as can be seen in figure 5.

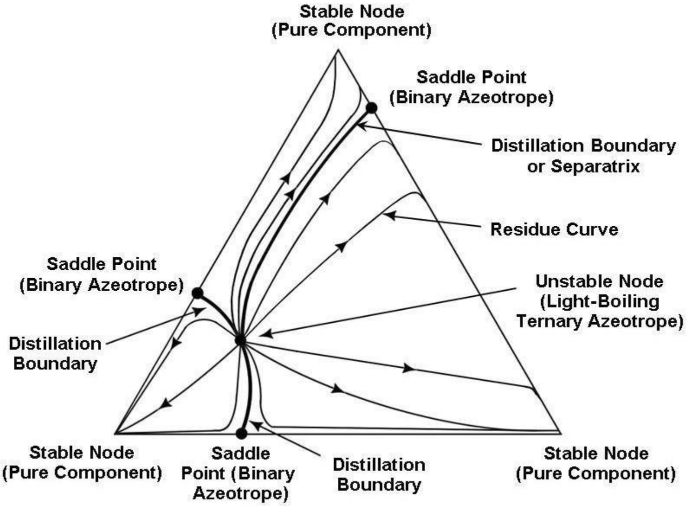


Figure 5: Elements of residue curve maps

(extracted image from Udeye et al. 2009. Fig 1)

In the vertices are the pure compounds or stable nodes; at that point, 100% of the mixture is the pure compound. The saddle point is a binary azeotrope formed by the compounds of the vertex. The unstable node corresponds to the ternary azeotrope. Residue curves are lines from the unstable node to the stable nodes. Distillation boundaries separate distillation regions, all residue curves inside the same area converge at the same point.

To study the feasibility of a process using residue curve maps is necessary to suppose a ∞/∞ analysis. This assumes an infinite reflux ratio and an infinite number of plates.

For a process to be feasible, it is necessary to check the feasibility of all distillation that forms it. The separation is possible if using the residue curve maps, three requirements are met simultaneously:

- First of all, a straight line connects the distillate, bottom, and feed.
- The second condition is that distillate and bottom must have a residue curve connecting them.
- Finally, the distillate profile and the bottom have a singular point (pure component or an azeotrope).

As said before, Aspen Plus has an option to generate residue curve maps. To do this, desired compounds must be introduced, select the thermodynamic model, go to Analysis, Residue Curves, and select the option that says: Use Distillation Synthesis ternary maps. Using this option, it is possible to find the azeotropes at a given pressure.

In this study, all the proposed processes operate at atmospheric pressure. Under these conditions, the residue curve map looks like the one in figure 6.

There are three differentiated regions. Temperature markings indicate where each azeotrope is located and its temperature. Closer inspection of figure 6 indicates that there are four azeotropes at atmospheric pressure: one ternary and three binaries. This should be considered when proposing viable processes.

Initially, it was thought that at four atmospheres, the ternary azeotrope would disappear, and some process proposals were made with this in mind. This can be seen in figure 7.

At first glance, it seems that there are only two azeotropes. But a closer inspection shows that all the residue curves start from the same point. This indicates the existence of an unstable node or a ternary azeotrope.

This fact makes it impossible for pressure variation to eliminate the ternary azeotrope. However, if in future adjustments of the thermodynamic model, with new experimental data, it is verified that there is not the ternary azeotrope, alternatives 4 and 5 could be considered.

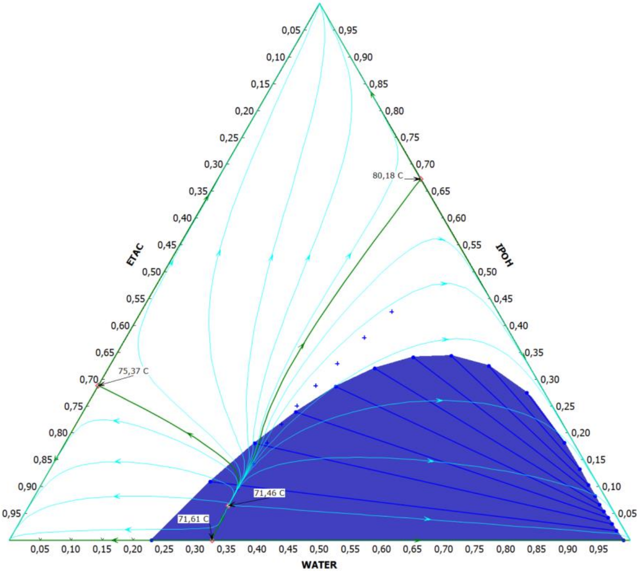


Figure 6: Residue curve map at atmospheric pressure

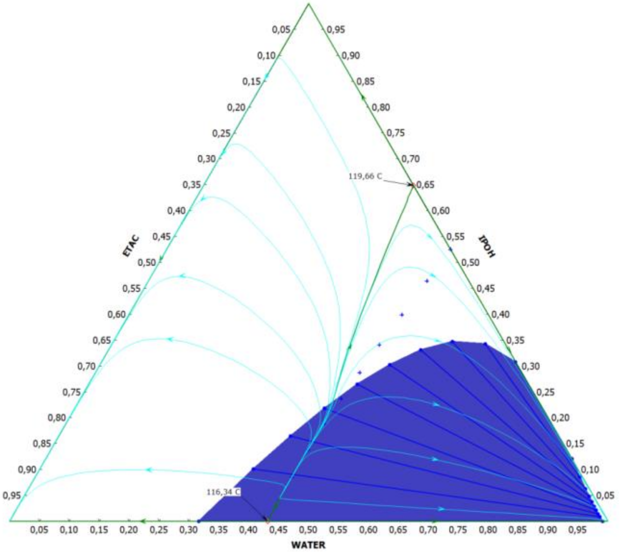


Figure 7: Residue curve map at four atmospheres

3.3. MASS BALANCES

As mentioned above, two programs have been used to calculate balances: Wolfram Mathematica and Xcas. Both have similar uses for this calculation. To solve a system of equations in Mathematica, it is done in the following way (Equation 2). Meanwhile in Xcas is equation 3.

$$Solve[eq1\ \&\&\ \ eq2\ \&\&\ eq3, \{var1, var2, var\}] \quad (2)$$

$$Solve([eq1, eq2, eq3], [var1, var2, va3\ ]) \quad (3)$$

In all balances, a feed of 100 kmol/h has been assumed. As for the composition of the compounds, those of the literature article have been used. These can be seen in table 4.

Table 4: Initial composition

Compound	Mass fraction	Mole fraction
Water	0.90	0.970
ETAC	0.08	0.025
IPOH	0.02	0.005

3.4. SHORTCUT SIMULATION

Once the balances have been resolved, it is necessary to carry out a first simple simulation. It is said to be simple because instead of using rigorous columns, separators are used. This type of simulation is carried out to check that the compounds are leaving the correct column and know the temperature.

Separators are used due to the use of the ∞/∞ model. The Sep2 (Figure 8) block is used to simulate the columns. Sep2 allows separating compounds by selecting the split fraction (or flow) and/or the mole (or mass) fraction.

As a separator, there is no temperature variation. Obtaining the temperature is necessary to calculate DSE, as will be seen in the following section.

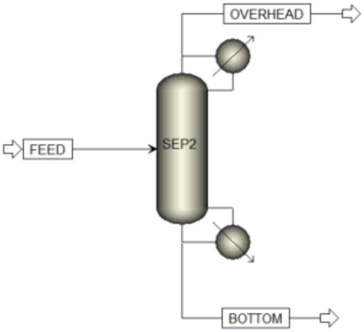


Figure 8 Sep2 block in Aspen Plus

To estimate the distillation and bottom temperature, it is necessary to add a heat exchanger. The “heater” block (Figure 9), which is located in the “exchanger” section, is used to do that. This block gives the temperature by entering the operating pressure and the vapour fraction of the mixture.

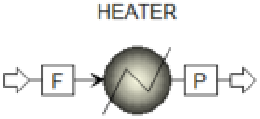


Figure 9: Heater block in Aspen Plus

3.5. DISTILLATION SEQUENCE EFFICIENCY (DSE)

Distillation Sequence Efficiency (DSE) is a parameter that contemplates the efficiency in the distillation columns. It is helpful to quantify and choose the most efficient processes in the early stages. It takes into account the molar flux rate, the feed stream and Carnot efficiency of each column in the process.

Efficiency also depends on the layout of the compounds and how many recirculations there are. The higher DSE and closer to unity, the more efficient the process will be. It can be negative in case of high recirculation flow.

It is an excellent indicator to discard processes that are not efficient and thus not make rigorous simulations.

DSE is based on ∞/∞ analysis, which implies that the investment and operational cost are infinite. This assumption prevents any cost assessment but allows to calculate of the Coefficient of Ease of Separation (CES), proposed by Nadgir and Liu et al. 1983:

$$CES = \frac{D}{B} \cdot (T_B - T_D) \quad (4)$$

Where D and B are distillate and bottom, respectively, and T_B and T_D are their temperatures.

ES allows knowing how easy it is to separate a mixture when the heuristics are contradictory. The problem with this parameter is that it is not suitable for the overall process. It is then necessary to use the DSE equation proposed by Plesu et al. 2015. This equation is a modification of equation 4, assuming the distillation columns as Carnot engines. DSE gives an overall distillation sequence efficiency.

$$DSE = \sum_i \frac{W_i}{F_c} \cdot \prod_C \eta_{ic} \quad (5)$$

W_i is the stream "i" depending on the feed, F_c the flow of crude feed and η_{ic} is the Carnot efficiency of the column.

Carnot efficiency describes the maximum thermal efficiency that a distillation column can achieve permitted by second thermodynamic law (all mechanical work can be transformed into heat, but not all heat can be transformed into mechanical work). The Carnot efficiency can be calculated according to equation 6.

$$\eta_{ic} = \frac{T_B - T_D}{T_B} \quad (6)$$

The following rules must be followed to calculate the DSE [11]:

- The bottom stream has a Carnot efficiency of 1.
- Per overhead column, the Carnot efficiency corresponds to that column.
- If there is a recirculation it is necessary to multiply the flow rate by the Carnot efficiency of the column minus 1.

Following these rules, the example in Figure 10 can be followed.

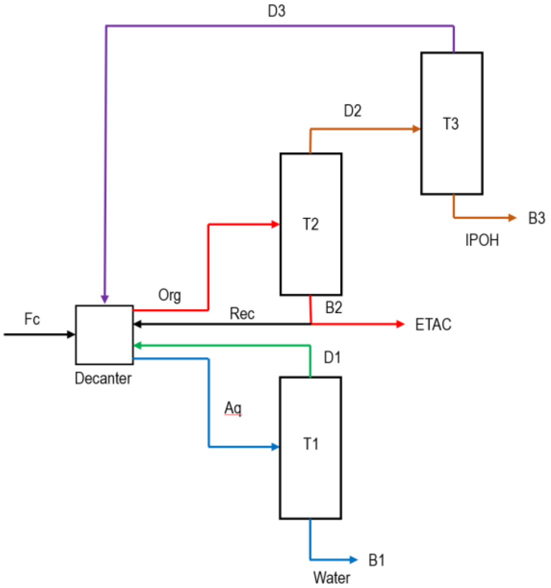


Figure 10: Representative example of DSE calculation

This example corresponds to propose alternative 1. The “Rec” stream is not painted because it moves forward with an efficiency of 1 but backwards with an efficiency of -1. This means that this stream does not influence the DSE. Equation 7 shows the DSE of this alternative.

$$DSE = \frac{B_1 + B_2 + B_3 \cdot \eta_2 + D_1 \cdot (\eta_1 - 1) + D_3 \cdot (\eta_2 \cdot \eta_3 - 1)}{F_C} \quad (7)$$

Carnot efficiency can be calculated by using equation 6, with the corresponding bottom and distillate temperatures. This process consists of three outlets and three recirculations. However, as mentioned above, recirculation of “Rec” is not taken into account.

The scheme will not be made for the rest of the proposed processes, but the equation will be put directly. The rest of the DSE equations and their results are shown in their respective section.

Once the DSEs of the processes have been calculated, a rigorous simulation can be carried out.

3.6. RIGOROUS SIMULATION

The main difference between the rigorous and the SEP simulations is that rigorous simulation serves to implement the process in reality. This implies that the simulation will be more complex, and more parameters will have to be considered.

The main block in rigorous simulation (talking about distillation) is the “RadFrac” block (Figure 11).

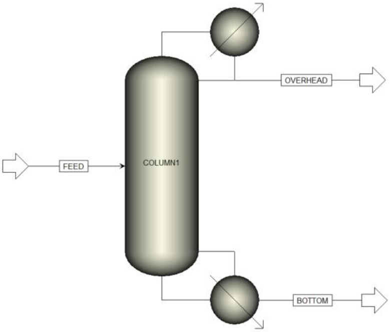
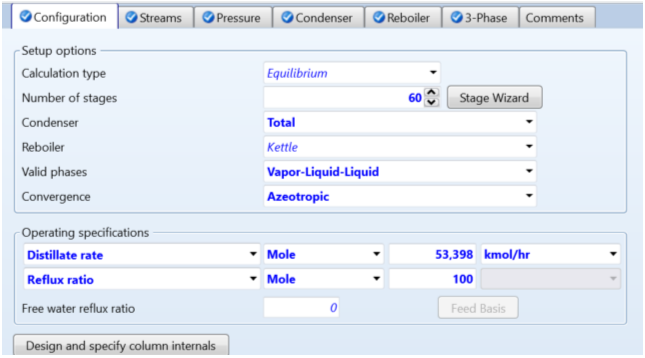


Figure 11:RadFrac block in Aspen Plus

As mentioned above, this block is more complex than SEP2. This can be seen by looking at the options it offers. Figure 12 shows the options offered by RadFrac.



Setup options
Calculation type: Equilibrium
Number of stages: 60
Condenser: Total
Reboiler: Kettle
Valid phases: Vapor-Liquid-Liquid
Convergence: Azeotropic

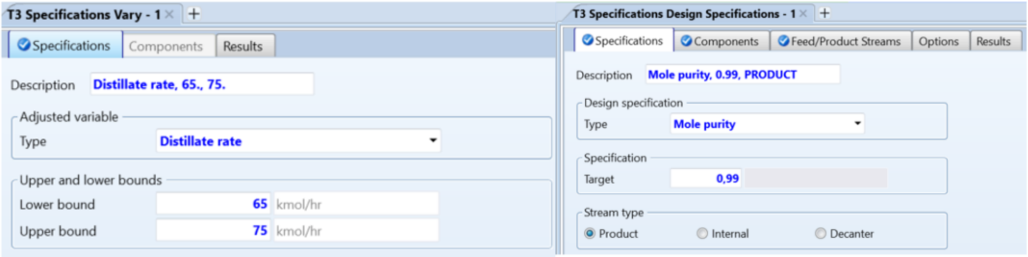
Operating specifications
Distillate rate: Mole 53,398 kmol/hr
Reflux ratio: Mole 100
Free water reflux ratio: 0

Figure 12: RadFrac options menu

Initially, it is necessary to assume the ∞/∞ analysis to ensure that the system converges. Therefore, enough stages have been selected to be considered infinite (60 stages). The same applies to the reflux ratio.

As rigorous simulation deals with a simulation of a real system, the convergence is set to azeotropic, and the valid phase is Vapour – Liquid – Liquid. Regarding the other options, in the "streams" section, a feed stage must be set, which usually is the middle plate (in the case of Figure 12, it is plate 30).

There are processes that are very sensitive to small variations in flow rate or composition. To obtain the required composition, it is necessary that the programme tend towards them. For this purpose, Aspen offers an option. To do that, in the block section, the column where are a sensitive problem, is selected. In "specifications" design specs and vary must be selected. Under "vary" it is selected that it should be varied to achieve the desired composition (in this case the distillation rate). On the other hand, under design spec, it is necessary to select the composition required (Figure 13).



Description: Distillate rate, 65., 75.

Adjusted variable

Type: Distillate rate

Upper and lower bounds

Lower bound: 65 kmol/hr

Upper bound: 75 kmol/hr

Description: Mole purity, 0.99, PRODUCT

Design specification

Type: Mole purity

Specification

Target: 0,99

Stream type

● Product ○ Internal ○ Decanter

Figure 13: Design specs and vary options in Aspen Plus

In rigorous simulation it is necessary to introduce more blocks to get close to reality. For the simulations of this study, mixers and splitters have been used (Figure 14). These are in the Mixers/Splitters section. These blocks are used to join or separate streams.

Figure 14: Mixer and Splitter blocks in Aspen Plus

Finally, decanters (Figure 15) have been used to separate part of the water from the organic fraction. Within Aspen these are in the separators tab.

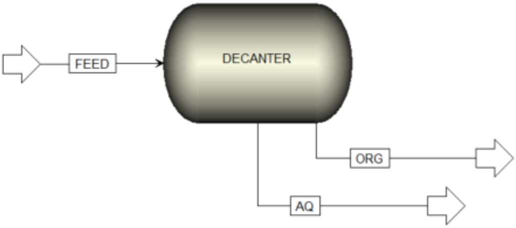


Figure 15: Decanter in Aspen Plus

Among the decanter options, an operating pressure must be selected. The other option to select is either the temperature or heat. In this case, the decanter is considered adiabatic, which implies that the temperature is the temperature of the distillate.

Once the process converges in the ∞/∞ analysis, the recirculations are switched on. At last, the optimisation is carried out. To do this, the number of plates and reflux ratio is reduced until an optimum number of stages and reflux is found.

4. RESULTS AND DISCUSSION

In this section, the results will be discussed for each process: The literature one (proposed by Hanwen Ge et. al. (2020) [3] and the proposed alternatives. The results will be shown following the steps described in the methodology.

4.1. PROPOSAL FROM THE LITERATURE

An outline of this process is given in figure 2 of the introduction section.

4.1.1. Residue curve map

This is a special case due to the existence of an extractive agent. That implies that the residue curve map will be more complex because of adding of a fourth compound. Therefore, the residue curve map is actually made up of 4 ternary 3D diagrams. Figure 16 shows the quaternary diagram.

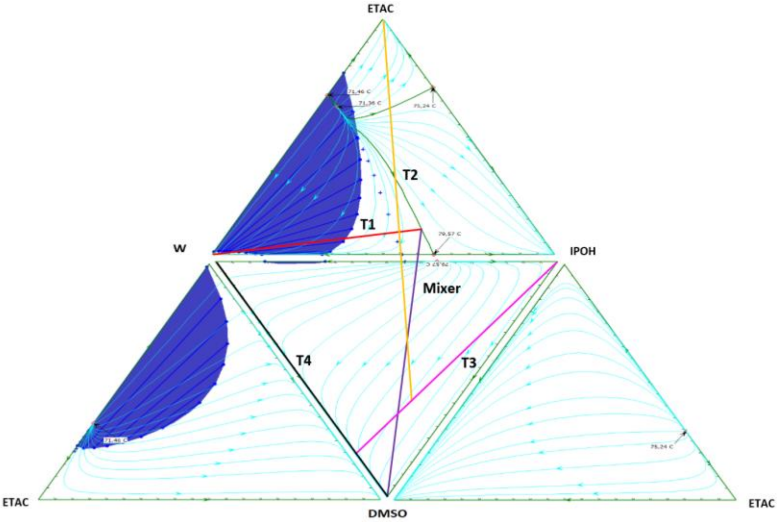


Figure 16: Literature setup quaternary diagram

As seen in the quaternary diagram, column 1 is fed by a ternary mixture of ETAC, IPOH and water. In this column, pure water is obtained from the bottom and distillate a mixture of ETAC, IPOH and, to a lesser extent, water.

D1 feeds column 2, in which DMSO (the extractive agent) is added. By affinity of the extractive agent with IPOH and water, ETAC is obtained from the distillate. Per bottom, a mixture of IPOH, DMSO and water is obtained.

The B2 stream feeds column 3. An auxiliary stream of DMSO enters this column. Due to the extractive agent has a higher affinity for water than IPOH, pure IPOH is obtained from the distillate. At the bottom, a binary mixture of water and DMSO is obtained.

At least, the binary mixture of DMSO and water is the feed for column 4. In this one, water is obtained from the distillate and DMSO from the bottom. To take advantage of the extractive agent, it is recirculated to columns 2 and 3.

4.1.2. Mass balances

As this is a process obtained from the literature, the results of the balances are already published in the article. Table 5 is a summary of the molar flow rate and composition of each stream.

Table 5A: Material balances of literature proposal

Stream	**Fc**	**D1**	**B1**	**D2**	**B2**	**DMSO**
Mole flow [kmol/h]	934.687	43.495	891.192	43.495	96.969	70.391
	Mole fraction					
Water	0.969	0.350	1	0	0.157	0
IPOH	0.026	0.555	0	0	0.249	0
ETAC	0.005	0.095	0	1	0.000	0
DMSO	0	0	0	0	0.594	1

Table 5B: Material balances of literature proposal

Stream	**D3**	**B3**	**D4**	**B4**
Mole flow [kmol/h]	24.153	143.207	15.2232	127.984
	Mole fraction			
Water	0	0.106	1	0
IPOH	1	0	0	0
ETAC	0	0	0	0
DMSO	0	0.894	0	1

In some streams, it is considered to be a pure compound, although, in reality, there are slight traces of others.

4.1.3. Shortcut simulation and DSE calculation

As explained in the methodology, once the balances have been obtained, a first simulation is carried out with SEPS in order to calculate the DSE. Figure 17 is the diagram simulated in Aspen plus. The auxiliary DMSO stream is not necessary because working in infinite analysis it is considered not to be lost.

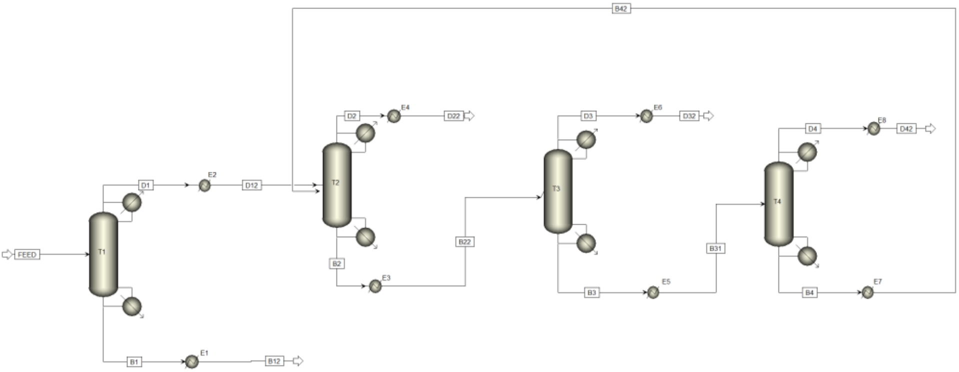


Figure 17: Literature proposal in Aspen Plus

The Carnot efficiencies for each column can be obtained. Table 6 illustrates the temperature and the efficiencies.

Table 6: Carnot efficiencies of proposal setup

Column 1		**Column 2**	
Tb1 [K]	372.8	Tb2 [K]	377.9
Td1 [K]	349.8	Td2 [K]	350.0
η1	0.062	η2	0.074
Column 3		**Column 4**	
Tb3 [K]	443.9	Tb4 [K]	463.4
Td3 [K]	354.9	Td4 [K]	371.8
η3	0.201	η4	0.198

All necessary data for the calculation of the DSE are obtained. The expression for the calculation of DSE for this configuration can be seen in equation 8.

$$DSE = \frac{B_1 + D_2 \cdot \eta_1 \cdot \eta_2 + D_3 \cdot \eta_1 \cdot \eta_3 + D_4 \cdot \eta_1 \cdot \eta_4}{F_C} \qquad (8)$$

. Solving this equation gives a DSE of 0.954. This is an elevated efficiency due to the recirculations are not considered. As this proposal is taken from an article[6], it is understood that it is possible to implemented, hence no rigorous simulation is required.

4.2. ALTERNATIVE 1

This first alternative (Figure 18) consists of one decanter and three columns. The crude feed is directly connected to the decanter, which is the primary process of this setup. In addition to being fed by the crude feed, the decanter is provided by the D3, Rec and D1 streams.

Two streams come out of the decanter: Aqueous and Organic streams. First, Aq directly feeds column 1. In this column, on the one hand, the pure water is separated (from the bottom), and, on the other hand, a mixture of water and IPOH is recirculated to the decanter (from the distillate).

On the contrary, the organic stream feeds column 2. In this column, ETAC is obtained per bottom. The B2 stream bifurcates into two streams, one into the decanter and in the other obtains pure ETAC. A mixture of water and IPOH is obtained from the distillate and feeds columns 3.

Pure IPOH is obtained in column 3 and a ternary azeotropic composition is recirculated to the decanter.

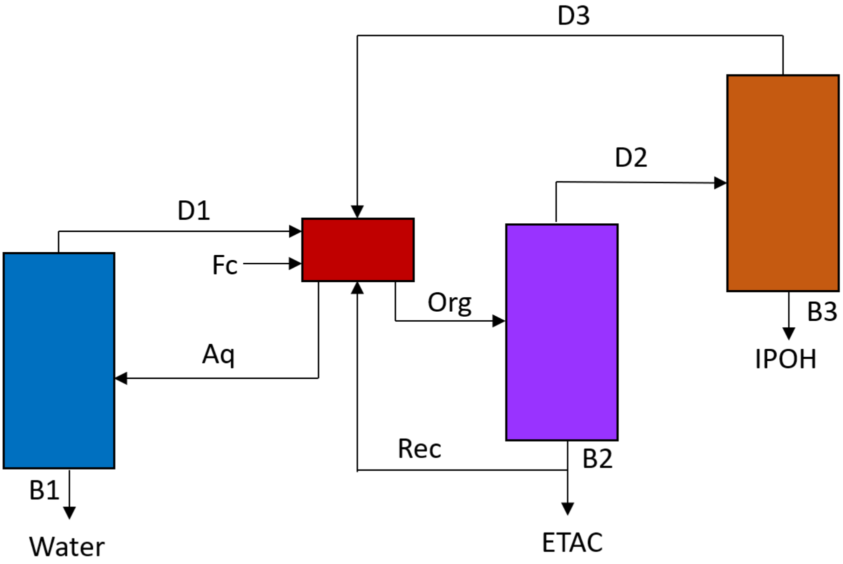


Figure 18: Alternative 1 setup

4.2.1. Residue curve map

Figure 19 illustrates the residue curve map. Each colour represents a column and the yellow dashed line corresponds to the recycle (Rec) stream.

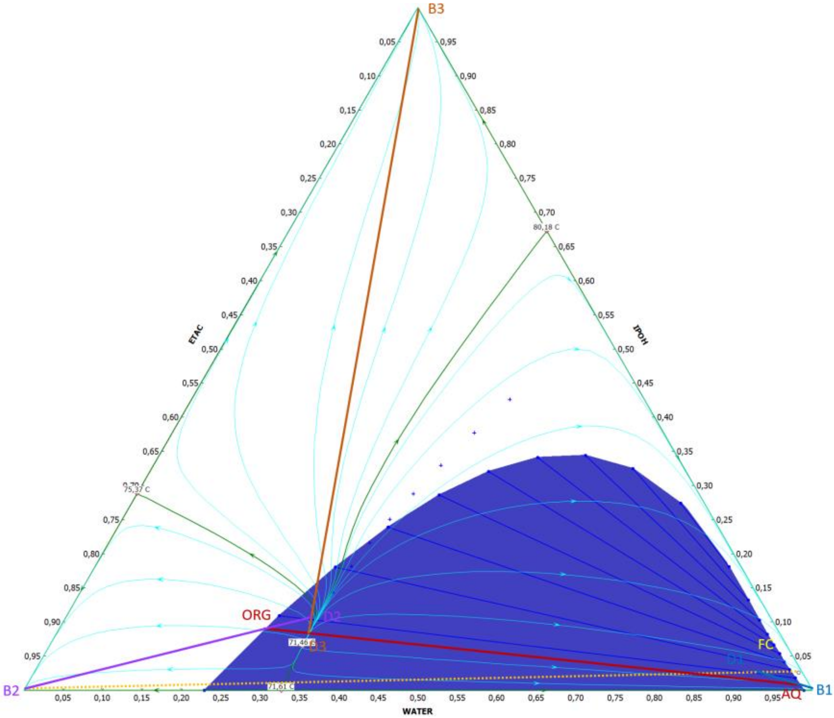


Figure 19: Residue curve map of Alternative 1

Following the feasibility rules explained in the methodology, it is checked that all rules are fulfilled. All columns are checked that distillate and bottom have a residue curve connecting them, a straight line connects distillate feed and bottom, and the profile between distillate and bottom has a singular point.

4.2.2. Mass balances

In this process, there is a degree of freedom due to the dependence between equations. It has been chosen to manipulate the water composition in distillate 1 because it has a restricted range. As shown in Figure 19, this composition can vary approximately plus/minus 0.2

What has been done is to look for a value of xd1w that gives the highest DSE, which has been 0.93. The following Table 7 summarizes the results of the material balances. The development of these and the material balances varying the composition are given in the appendix.

Table 7A: Material balance of alternative 1 (degree of freedom xd1w = 0.93)

Stream	**Fc**	**Org**	**Aq**	**D1**	**B1**	**D2**	**B2**
Mole flow [kmol/h]	100	87.772	151.331	30.124	97.933	70.796	16.977
	Mole fraction						
Water	0.970	0.253	0.976	0.930	0.990	0.314	0
IPOH	0.026	0.071	0.012	0.017	0.010	0.086	0.010
ETAC	0.004	0.675	0.012	0.053	0	0.600	0.990

Table 7B: Material balance of alternative 1 (degree of freedom xd1w = 0.93)

Stream	**D3**	**B3**	**Rec**	**ETAC**
Mole flow [kmol/h]	69.183	1.612	16.522	0.455
	Mole fraction			
Water	0.321	0.01	0.000	0.000
IPOH	0.065	0.99	0.010	0.010
ETAC	0.614	0	0.990	0.990

This is a system of 13 equations, 13 unknowns and 1 degree of freedom. Column 2 and 3 and decanter mass balances have been used. Column 1 mass balances are linear combination of the other unit mass balances plus the overall balances.

4.2.3. DSE calculation

In this case, a rigorous simulation has been carried out. For this reason, a figure of the process with SEPS is not attached. The Carnot efficiencies, such as the temperatures, are shown in table 8.

Table 8: Temperature and Carnot efficiencies alternative 1

Column 1		**Column 2**	
Tb1 [K]	372.8	Tb2 [K]	350.0
Td1 [K]	344.2	Td2 [K]	344.2
η1	0.077	η2	0.017
Column 3			
Tb3 [K]	354.9		
Td3 [K]	343.9		
η3	0.031		

The DSE is then calculated using equation 9. The Rec stream does not influence the DSE since it moves backwards and forwards with -1 and 1.

$$DSE = \frac{B_1 + B_2 + B_3 \cdot \eta_2 + D_1 \cdot (\eta_1 - 1) + D_3 \cdot (\eta_2 \cdot \eta_3 - 1)}{Fc} \quad (9)$$

This calculation results in a DSE of 0.179. It is an acceptable DSE because it is a more complex system with several recirculations.

4.2.4. Rigorous simulation

Figure 20 shows the rigorous simulation of alternative 1 and mass balance obtained in Aspen. Comparing the balances in Table 7 with those obtained by simulation, they are practically the same. This confirms that the process can be carried out [15].

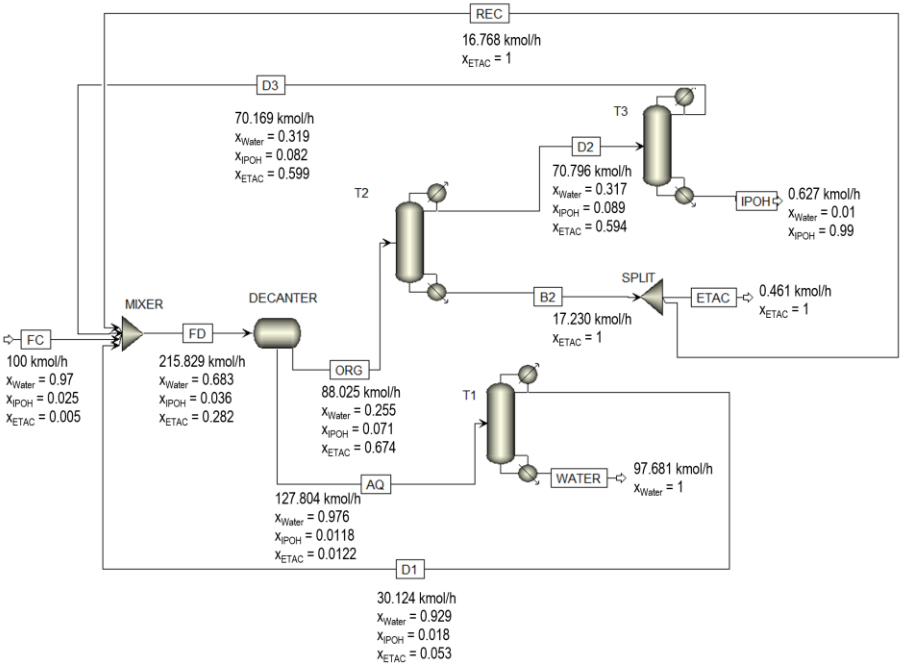


Figure 20: Rigorous simulation setup 1

This process is susceptible to variations in composition and flow rate. A slight variation of these leads to different results. Figure 21 shows how a slight variation of the flow rate D3, significantly changes the bottom composition of column 3. This is due to the proximity between the feed, F3, and the distillate D3.

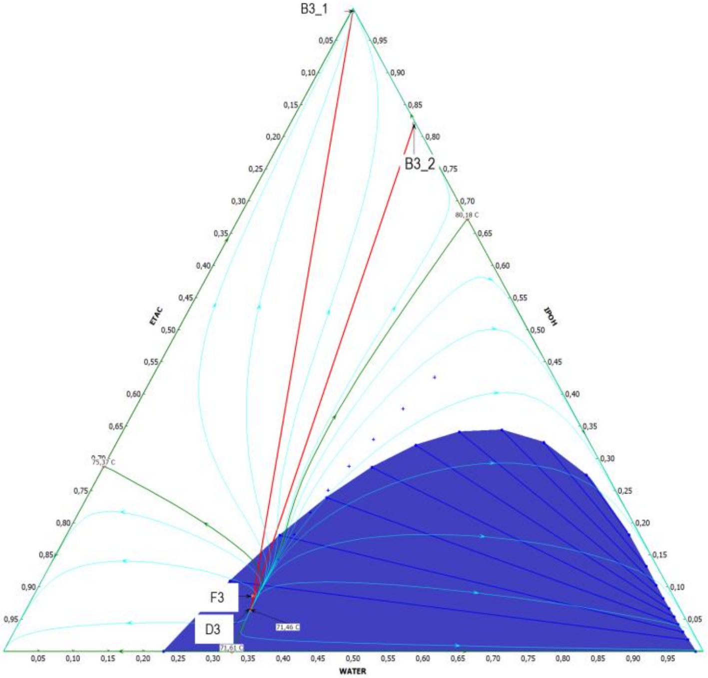
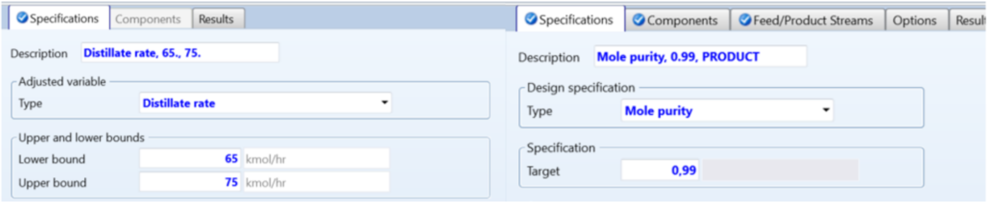


Figure 21: Sensitivity in column 3

Where F3 is D2 in figure 20 and B3_1, and B3_2 are the two possible compositions in this system. As can be seen with D3 and F3 fixed, it is possible to obtain two possible results at the bottom of the column. The desired multiplicity is B3_1, which corresponds to pure IPOH. To force to get B3_1, as mentioned above, it is necessary to use the “Vary” and “Design Spec” option in column 3. Figure 22 shows the configuration to obtain pure IPOH.



Description: Distillate rate, 65., 75.

Adjusted variable
Type: Distillate rate

Upper and lower bounds
Lower bound: 65 kmol/hr
Upper bound: 75 kmol/hr

Description: Mole purity, 0.99, PRODUCT

Design specification
Type: Mole purity

Specification
Target: 0,99

Figure 22: Vary and Design Spec of column 3

Under vary (left picture), a range between 65 and 75 kmol/h is set. Although in the material balance, the result of D3 was 69, it is necessary to have a range to obtain that composition. Meanwhile, in Design Spec, a 0.99 composition of IPOH in the IPOH stream is fixed.

Table 9A and 9B shows the results obtained for the composition, molar flow, and temperature of each stream in the simulation.

Table 9A: Simulation results of alternative 1

Stream	**Fc**	**Org**	**Aq**	**D1**	**B1**	**D2**
Temperature[K]	353.970	344.985	344.985	344.698	372.799	344.246
Mole flow[kmol/h]	100	88.025	127.804	30.124	97.681	70.796
	Mole fraction					
Water	0.970	0.255	0.976	0.929	1.000	0.317
IPOH	0.025	0.071	0.012	0.018	0.000	0.089
ETAC	0.005	0.674	0.012	0.053	0.000	0.594

Table 9B: Simulation results of alternative 1

Stream	**B2**	**D3**	**B3**	**ETAC**	**Rec**	**Fd**
Temperature [K]	349.954	344.238	354.745	349.954	349.954	341.648
Mole flow[kmol/h]	17.230	70.169	0.627	0.461	16.768	215.829
	Mole fraction					
Water	0.000	0.319	0.010	0.000	0.000	0.683
IPOH	0.000	0.082	0.990	0.000	0.000	0.036
ETAC	1.000	0.599	0.000	1.000	1.000	0.282

As mentioned above, the simulation balances are practically the same as expected. This confirms that the implementation is feasible.

4.3. ALTERNATIVE 2

Alternative 2 (Figure 23) is a bit more complex than alternative 1. It consists of 4 columns and 1 decanter. Crude feed is mixed with distillate D4 to feed column 1. This column separates water from the bottom and a ternary mixture from the distillate.

D1 is mixed with D3 to feed column 2. From distillate, a ternary azeotropic composition is providing the decanter. From the bottom, a binary azeotropic composition of water and IPOH feed column 4.

In the decanter, more water is extracted, and an organic phase fed to column 3. In this column, pure ETAC is obtained at the bottom and distillate is recirculated to D1. In column 4 pure IPOH is obtained, while the distillate is mixed with the crude aliment.

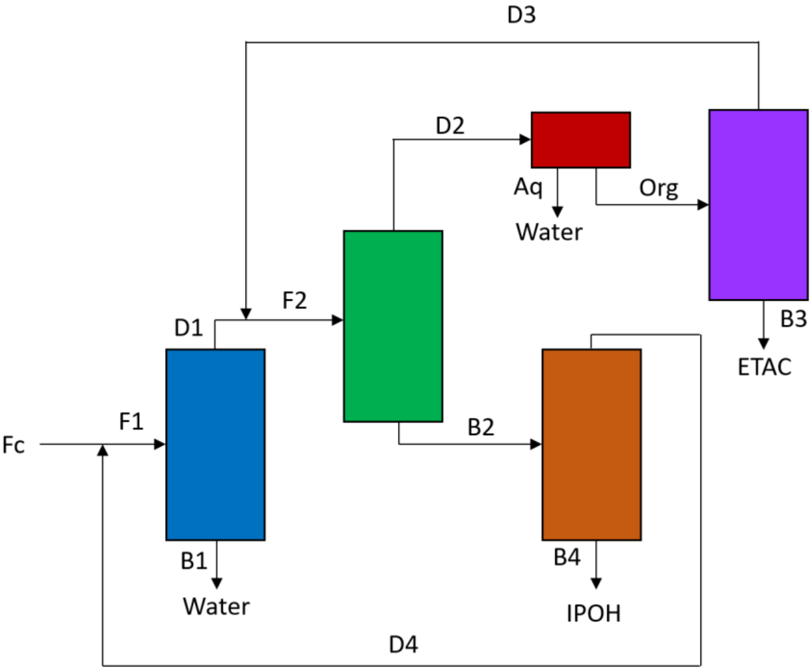


Figure 23: Alternative 2 setup

4.3.1. Residue curve map

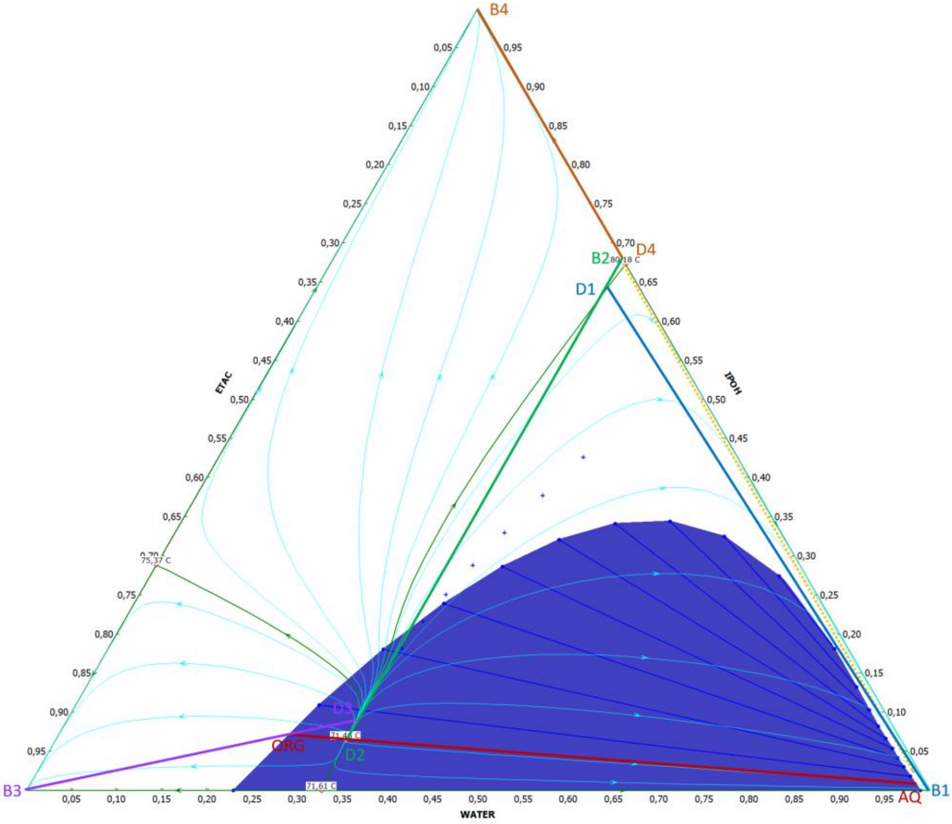


Figure 24: Residue curve map of alternative 2

As in the previous case, each colour represents a column. Figure 24 shows the residue curve maps of the alternative 2.

The highlights of this residue curve map are the compositions in column 2 (straight orange line). The distillate in the column corresponds to the ternary azeotropic composition. At the same time, the bottom is approximately the composition of the binary azeotrope formed by water – isopropyl alcohol.

As the binary azeotrope cannot be taken out directly, the implementation of column 4 (straight green line) is necessary to achieve pure IPOH. The distillate from this column is mixed with the crude feed to feed column 1.

4.3.2. Mass balances

As in the previous case, there is one degree of freedom. In this case, the composition of the IPOH at the bottom of column 2 (xb2ip) has been chosen. It has little range of variation because it is at the limit of the distillation region. To choose the most optimal composition it is necessary to check which gives the best DSE. In this case, the best xb2ip is 0.673. Balances with other compositions can be found in the appendix.

In addition, this process has a special condition: The distillate from column 1 must be located in the upper distillation region. To do this, points were taken along the curve of the distillation region and the equation of the curve was obtained (Equation 10).

$$\mathrm{xd1w} = -1.495 \cdot \mathrm{xd1ip}^3 + 2.3769 \cdot \mathrm{xd1ip2}^2 - 1.0989 \cdot \mathrm{xd1ip} + 0.4474 \quad (10)$$

Another condition is required. The distillate from column 3 must be in the straight line of the ternary azeotrope. This equation can be seen in equation 11:

$$\mathrm{xd3w} = -0.1034 \cdot \mathrm{xd3ip} + 0.3294 \quad (11)$$

Taking all this into account, the balances were solved, and the results shown in Table 10 were obtained:

Table 10 A: Material balances of alternative 2 (degree of freedom xb2ip = 0.673)

Stream	**Fc**	**F1**	**F2**	**Org**	**Aq**	**D1**	**B1**
Mole flow [kmol/h]	100	223.490	129.313	3.131	0.309	126.810	96.681
	Mole fraction						
Water	0.970	0.615	0.328	0.256	0.977	0.328	0.990
IPOH	0.026	0.383	0.657	0.070	0.011	0.668	0.010
ETAC	0.004	0.002	0.015	0.673	0.012	0.004	0

Table 10 B: Material balances of alternative 2 (degree of freedom xb2ip = 0.673)

Stream	**D2**	**B2**	**D3**	**B3**	**D4**	**B4**
Mole flow [kmol/h]	3.440	125.873	2.503	0.628	123.490	2.383
	Mole fraction					
Water	0.321	0.327	0.321	0	0.327	0.010
IPOH	0.065	0.673	0.085	0.010	0.673	0.99
ETAC	0.614	0	0.594	0.990	0.000	0

The development of the balances can be found in the appendix. This is a system of 20 unknowns, 20 equations and 1 degree of freedom.

4.3.3. DSE calculation

To obtain the DSE is necessary to make the first simulation with SEPS. At first glance, it is identical but without the heat exchangers. However, in terms of simulation, it is simpler than the rigorous one. Table 11 presents the Carnot efficiencies and temperatures of this process. With all this it is now possible to calculate the DSE as shown in equation 12:

Table 11: Carnot efficiencies and temperatures. Alternative 2

Column 1		**Column 2**	
Tb1 [K]	372.8	Tb2 [K]	353.7
Td1 [K]	353.6	Td2 [K]	343.5
η1	0.052	η2	0.029
Column 3		**Column 4**	
Tb3 [K]	350.0	Tb4 [K]	354.9
Td3 [K]	344.3	Td4 [K]	353.7
η3	0.016	η4	0.003

$$DSE = \frac{B_1 + B_3 \cdot \eta_1 \cdot \eta_2 + B_4 \cdot \eta_1 + Aq \cdot \eta_1 \cdot \eta_2 + D_3 \cdot (\eta_2 \cdot \eta_3 - 1) + D_4 \cdot (\eta_1 \cdot \eta_4 - 1)}{Fc} \quad (12)$$

Solving the equation gives a DSE of -0,292. This is a very low value, which is since there is a very high recirculated flow. This causes efficiency to drop drastically.

4.3.4. Rigorous simulation

Figure 25 presents the rigorous simulation of this alternative. As can be seen, the balances coincide with Table 10. This means that it can be extrapolated to reality [15].

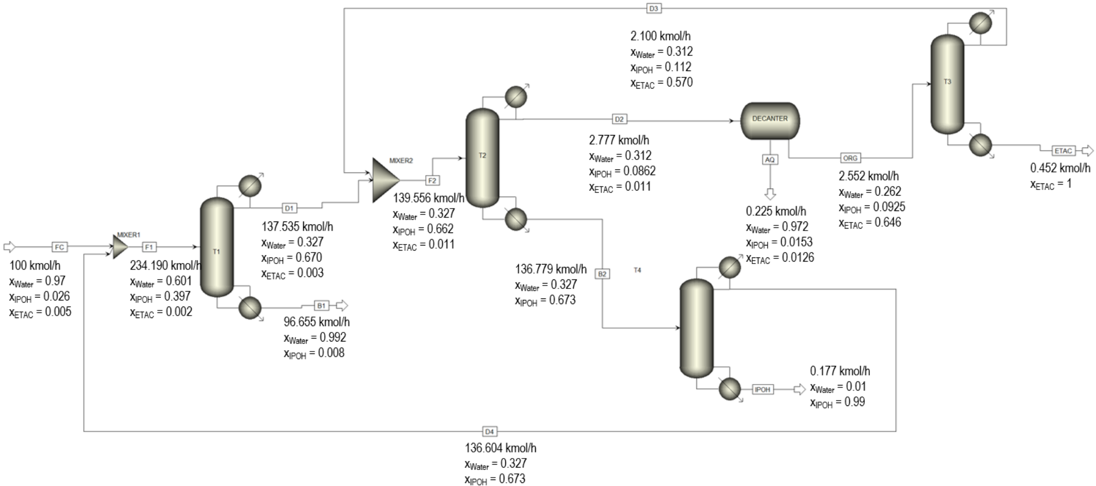


Figure 25: Rigorous simulation Alternative 2

No multiplicity problems occur in this case. Therefore, the process was simulated without any issues. However, a design spec was made to ensure that the output compositions are as expected. The same was done as in Figure 22, but with the relevant values of this process.

Tables 12A and 12B presents the values (composition, molar flow, and temperature) obtained for each stream in the simulation 2.

Table 12A: Simulation results of alternative 2

Stream	**Fc**	**F1**	**F2**	**Org**	**Aq**	**D1**	**B1**
Temperature [K]	353.970	353.530	352.371	344.244	344.244	352.893	368.187
Mole flow[kmol/h]	100	234.190	139.556	2.552	0.225	137.535	96.655
	Mole fraction						
Water	0.970	0.601	0.327	0.262	0.972	0.327	0.992
IPOH	0.026	0.397	0.662	0.093	0.015	0.670	0.008
ETAC	0.004	0.020	0.011	0.646	0.013	0.003	0

Table 12B: Simulation results of alternative 2

Stream	**D2**	**B2**	**D3**	**B3**	**D4**	**B4**
Temperature [K]	344.244	353.003	344.292	349.954	353.003	354.745
Mole flow[kmol/h]	2.777	125.873	2.100	0.452	136.604	0.177
	Mole fraction					
Water	0.312	0.327	0.312	0	0.327	0.010
IPOH	0.0862	0.673	0.112	0	0.673	0.99
ETAC	0.602	0	0.576	1.000	0	0

As was the case in alternative 1, the balances obtained from the simulation are close to those expected.

4.4. **ALTERNATIVE 3**

The last alternative (Figure 26) is the most complex of the above. This is since it has the most recirculation, which makes it difficult to simulate and design. The system consists of 3 columns, where the pure compounds will be obtained, and 1 decanter.

Crude feed is mixed with the aqueous stream to feed column 1. In this column, it is possible to obtain pure water from the bottom and a ternary mixture from the distillate. The distillation steam of column 1 (D1) will be mixed with a fraction of organic steam (in figure 26 it is S stream).

The mixture of S and D1 streams is obtained F2, one of the feeds of column 2. The other supply stream of column 2 is D3. D3 comes from column 3. This is fed with F3 steam, which is a part of the unmixed Org stream. Column 3 gives D3, which is a mixture close to the ternary azeotrope composition, and pure ETAC at the bottom.

Finally, column 2 is fed by D3 and F2. This column distillate the ternary azeotrope that feeds the decanter, while pure IPOH is obtained at the bottom.

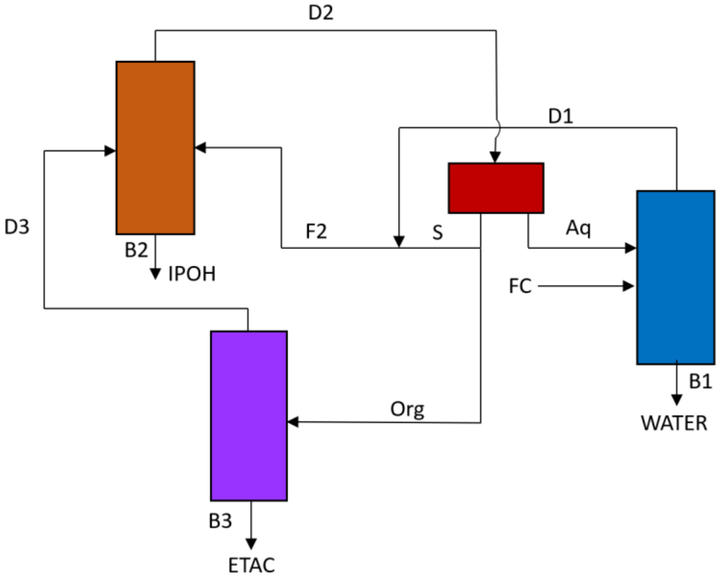


Figure 26: Alternative 3 setup

4.4.1. Residue curve map

Figure 27 presents the residue curve map of alternative 3. Each colour represents a column in Figure 26.

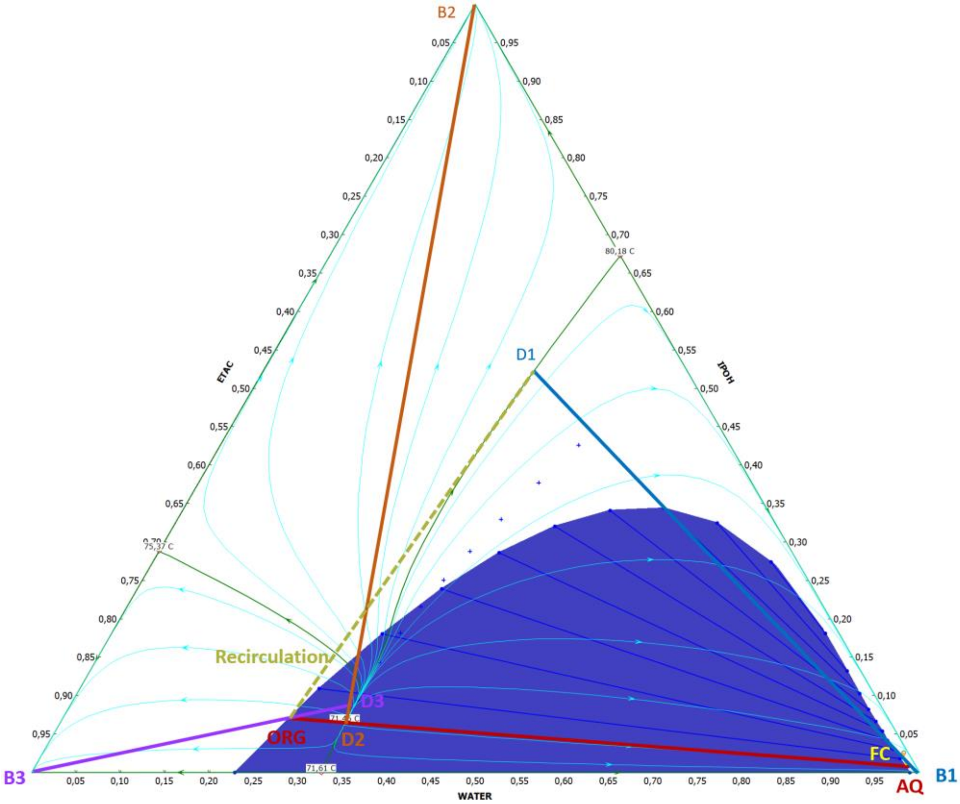


Figure 27: Residue curve map of alternative 3

The most remarkable thing about this residue curve map is the dashed purple line. This line means that D1 is mixed with the Org stream to feed column 2 (greed line). That point may confuse because the Org stream also provides column 3 (red line). For this case, add a split for a part of Org, feed the column, and mix with D1.

4.4.2. Mass balances

This system is completely defined. This means that there are no degrees of freedom. Thus, there is only one solution. This case also has a special condition: D1 must be on the straight line (not on the curve) of the distillation region (Equation 13). The other is that the ratio xd3w/xd3etac must have the same ratio as the ternary azeotropic composition, for it to be located at that point (Equation 14). The ternary azeotropic composition can be seen in Table 2.

$$\mathrm{xd1ip} = 8.5618 \cdot \mathrm{xd1w} - 2.086 \quad (13)$$

$$\frac{0.321}{0.614} = \frac{xd3w}{xd3etac} = \frac{xd3w}{1 - xd3w - xd3ip} \quad (14)$$

In equation 12, xd3etac is decomposed using the definition of the molar fraction to have in the denominator xd3w and xd3ip. These due to the fact that in the material balances, these two have been used and not the ETAC composition.

As in the other cases, the developed material balances are in the appendix. Table 13 presents the results of these.

Table 13 A: Material balances of alternative 3

Stream	**Fc**	**F2**	**F3**	**Org**	**Aq**	**S**
Mole flow[kmol/h]	100	9.311	2.740	9.115	0.900	6.375
	Mole fraction					
Water	0.970	0.272	0.256	0.256	0.976	0.256
IPOH	0.025	0.213	0.071	0.071	0.011	0.071
ETAC	0.005	0.516	0.673	0.673	0.013	0.673

Table 13 B: Material balances of alternative 3

Stream	**D1**	**B1**	**D2**	**B2**	**D3**	**B3**
Mole flow[kmol/h]	2.936	97.964	10.015	1.531	2.235	0.505
	Mole fraction					
Water	0.305	0.990	0.321	0.010	0.314	0
IPOH	0.521	0.010	0.065	0.990	0.085	0.010
ETAC	0.174	0	0.614	0	0.601	0.990

4.4.3. DSE calculation

As in the other alternatives, the simulation scheme with SEPS is not presented inasmuch it is very similar to the rigorous simulation scheme. In any case, this has been done to obtain the Carnot efficiencies, which are shown in Table 14.

Table 14: Carnot efficiencies and temperatures of setup 3

Column 1		**Column 2**	
Tb1 [K]	372.8	Tb2 [K]	354.9
Td1 [K]	349.1	Td2 [K]	344.0
η1	0.064	η2	0.031
Column 3			
Tb3 [K]	350.0		
Td3 [K]	344.8		
η3	0.015		

The expression to obtain the DSE for this alternative is presented in equation 15:

$$DSE = \frac{B_1 + B_2 \cdot \eta_1 \cdot \eta_2 \cdot \eta_3 + B_3 \cdot \eta_2 \cdot \eta_3 + D_1 \cdot (\eta_1 - 1) + D_3 \cdot (\eta_2 \cdot \eta_3 - 1)}{Fc} \qquad (15)$$

Solving this equation gives a DSE of 0,929. It is a very high DSE for the reason that the recirculated streams flow has a very small flow rate compared to the other streams. This results in a small negative impact on efficiency.

4.4.4. Rigorous simulation

Figure 28 presents the rigorous simulation scheme of the third alternative. The recirculated flows are small compared to the others. Therefore, the DSE is high. As in the other alternatives, the simulation mass balances results are similar to the theoretical ones [15].

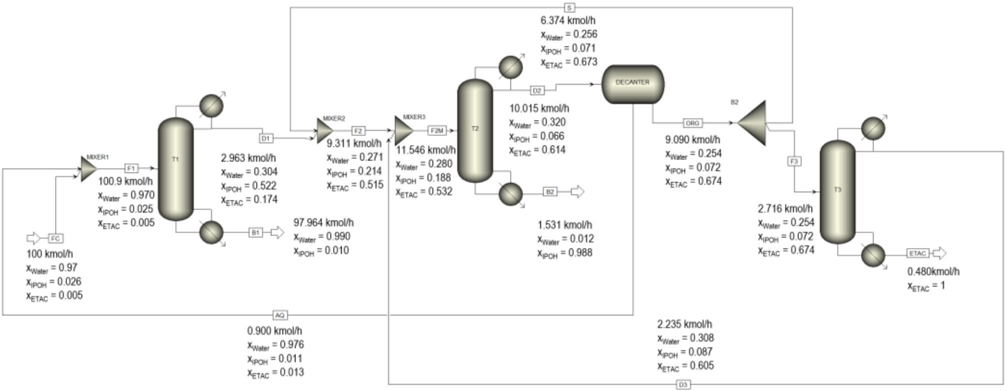


Figure 28: Rigorous simulation of alternative setup 3

Tables 15A and 15B presents the values (composition, molar flow, and temperature) obtained for each stream in the simulation 3.

Table 15A: Simulation results of alternative 3

Stream	**FC**	**F1**	**F2**	**F3**	**Org**	**Aq**	**D1**
Temperature [K]	353.275	353.183	342.233	344.230	344.230	344.230	348.485
Mole flow[kmol/h]	100	100.900	9.311	2.716	9.090	0.900	2.963
	Mole fraction						
Water	0.970	0.970	0.271	0.254	0.254	0.976	0.304
IPOH	0.025	0.025	0.214	0.072	0.072	0.011	0.522
ETAC	0.005	0.005	0.515	0.674	0.674	0.013	0.174

Table 15B: Simulation results of alternative 3

Stream	**B1**	**D2**	**B2**	**D3**	**B3**	**S**	**F2M**
Temperature [K]	367.079	344.230	354.710	344.244	349.954	344.230	342.463
Mole flow[kmol/h]	97.964	10.015	1.531	2.235	0.480	6.374	11.546
	Mole fraction						
Water	0.99	0.320	0.012	0.308	0.000	0.256	0.28
IPOH	0.01	0.066	0.988	0.087	0.000	0.071	0.188
ETAC	0.000	0.614	0.000	0.605	1.000	0.673	0.532

As in the other alternatives, it is also verified that the expected balances are practically the same as the simulated ones.

4.4.5. Variant of third alternative

Alternative 3 has a variant, where the feed in column 3 instead of being fed directly from the organic stream, is a mixture of the organic stream and distillate 1. The schematic of this variant is shown in Figure 29.

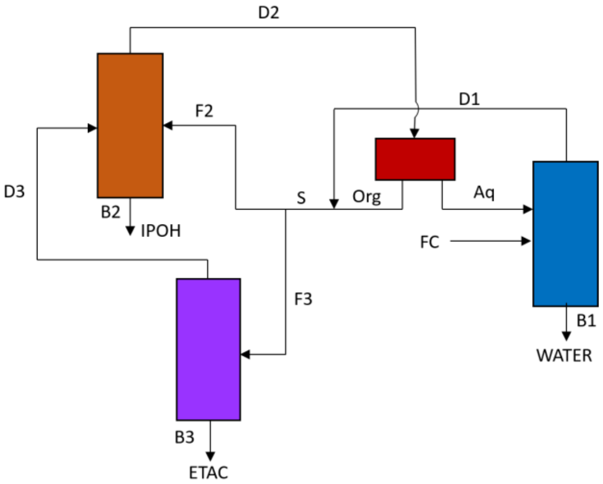


Figure 29: Alternative 3 variant setup

4.4.5.1. *Mass balances and DSE calculation*

The mass balances differ from alternative 3 because S and F3 streams are a mixture of the organic distillate 1 as shown in Table 16.

Table 16 A: Material balances of alternative 3 variant

Stream	Fc	F2	F3	Org	Aq	S
Mole flow[kmol/h]	100	0.001	29.107	24.638	2.434	29.108
	Mole fraction					
Water	0.970	0.265	0.265	0.256	0.976	0.265
IPOH	0.025	0.147	0.147	0.071	0.011	0.147
ETAC	0.005	0.588	0.588	0.673	0.013	0.588

Table 16 B: Material balances of alternative 3 variant

Stream	D1	B1	D2	B2	D3	B3
Mole flow[kmol/h]	4.469	97.964	27.072	1.531	28.602	0.505
	Mole fraction					
Water	0.316	0.990	0.321	0.010	0.304	0
IPOH	0.565	0.010	0.065	0.990	0.115	0.010
ETAC	0.119	0	0.614	0	0.581	0.990

The equation for calculating the DSE and its value is different from alternative 3 due to the change discussed above. The values of the temperatures and Carnot efficiencies of this variant are the same as for alternative 3, presented in Table 14. The expression of the DSE is different, as shown in equation 16.

$$DSE = \frac{B_1 + B_2 \cdot \eta_1 \cdot (y \cdot \eta_2 + x \cdot \eta_3) + B_3 \cdot \eta_1 \cdot \eta_2 \cdot \eta_3 + D_1 \cdot (\eta_1 - 1) + D_3 \cdot (\eta_2 \cdot \eta_3 - 1)}{Fc} \quad (16)$$

Solving this equation gives a DSE of 0.652. It is a lower value than alternative 3 because of the amount of recirculated flow (D1 and D3 streams) in the variant are considerably higher than in alternative 3. In addition, in column 2, a factor must be added to know the influence of column 3 and column 2. X and Y are the percentage of the flows of "S" stream that is split into the streams F2 and F3. This is shown in equation 17 and 18.

$$x = \frac{F3}{S} \quad (17)$$

$$y = 1 - x \quad (18)$$

Calculating these equations, x and y gives that "x" represents the 99% of the flow coming from S while "y" represents 1%.

4.4.5.2. Rigorous simulation

The particularity of this variant (Figure 30) is that there is the phenomenon of multiplicities in column 2. As mentioned in the Multiple Steady States section, with the same feed, number of stages, feed stage, and the exact bottom and distillate rate, different compositions of distillate and bottom are obtained [15].

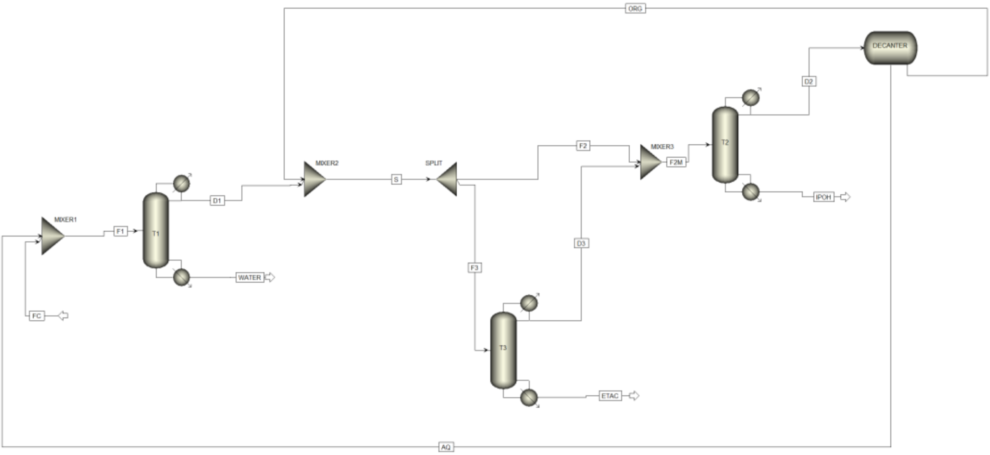


Figure 30: Rigorous simulation of alternative 3 variant setup

4.4.5.3. Multiple Steady States

As discussed above, in column 2, there are multiplicities. Tables 17 and 18 presents the flow rate, temperatures, and compositions of the two possible multiplicities obtained in the simulation of the variant.

Table 17 A: Binary azeotrope multiplicity simulation data

Stream	**Fc**	**Org**	**Aq**	**D1**	**D2**	**D3**	**B1**
Temperature [K]	176.225	159.936	158.667	169.639	160.126	160.156	211.368
Mole flow [kmol/h]	100	24.638	2.434	4.469	25.000	25.752	97.964
	Mole fraction						
Water	0.970	0.256	0.976	0.316	0.309	0.300	1.000
IPOH	0.025	0.071	0.011	0.565	0.128	0.135	0
ETAC	0.005	0.673	0.013	0.119	0.563	0.565	0

Table 17 B: Binary azeotrope multiplicity simulation data

Stream	**B2**	**B3**	**F2**	**F3**	**S**	**F2M**
Temperature [K]	167.100	167.023	156.962	156.962	156.962	159.296
Mole flow [kmol/h]	0.753	3.355	0.001	29.107	29.108	25.753
	Mole fraction					
Water	0.000	0.000	0.265	0.265	0.265	0.300
IPOH	0.299	0.240	0.147	0.147	0.147	0.135
ETAC	0.701	0.760	0.588	0.588	0.588	0.565

As shown in Tables 17 A and 17 B, the balances in most of the flows coincide with those expected. Those that differ are those in column 2, due to Aspen found the multiplicity that was not desired.

Table 18 A: IPOH multiplicity simulation data

Stream	**Fc**	**Org**	**Aq**	**D1**	**D2**	**D3**	**B1**
Temperature [K]	353.275	344.226	343.521	349.616	344.236	344.348	372.799
Mole flow [kmol/h]	100	24.638	2.434	4.469	24.163	25.752	97.964
	Mole fraction						
Water	0.970	0.256	0.976	0.316	0.320	0.300	1.000
IPOH	0.025	0.071	0.011	0.565	0.078	0.135	0.000
ETAC	0.005	0.673	0.013	0.119	0.602	0.565	0.000

Table 18 B: IPOH multiplicity simulation data

Stream	**B2**	**B3**	**F2**	**F3**	**S**	**F2M**
Temperature [K]	354.672	348.163	342.574	342.574	342.574	343.870
Mole flow [kmol/h]	1.590	3.355	0.001	29.107	29.108	25.753
	Mole fraction					
Water	0.000	0.000	0.265	0.265	0.265	0.300
IPOH	0.990	0.240	0.147	0.147	0.147	0.135
ETAC	0.010	0.760	0.588	0.588	0.588	0.565

Figure 31 illustrate the two multiplicities of column 2 in the residue curve maps. As can be seen in Tables 17 and 18, both have the same composition and molar flow rate of column 2 feed (F2M).

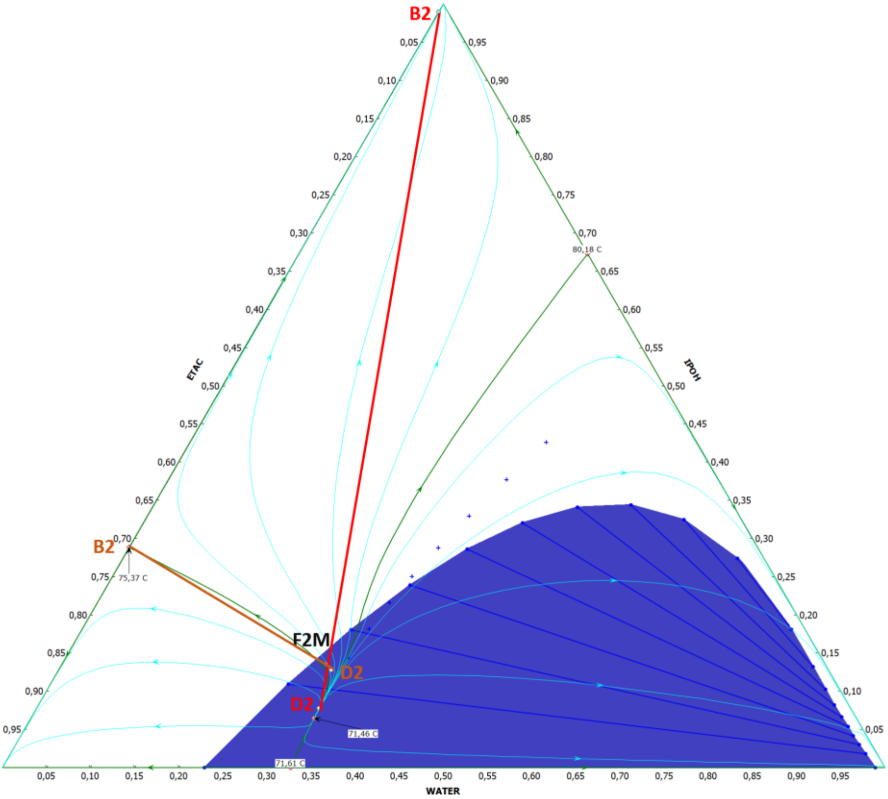


Figure 31: Multiplicities in column 2

4.4.6. Composition profiles and economic costs

As this is the alternative with the highest DSE of all the alternative, a more in-depth study is carried out. Figures 32, 33 and 34 below present the composition profile of each compound with the number of stages in each column of alternative 3.

Figure 32: Composition profiles of Column 1

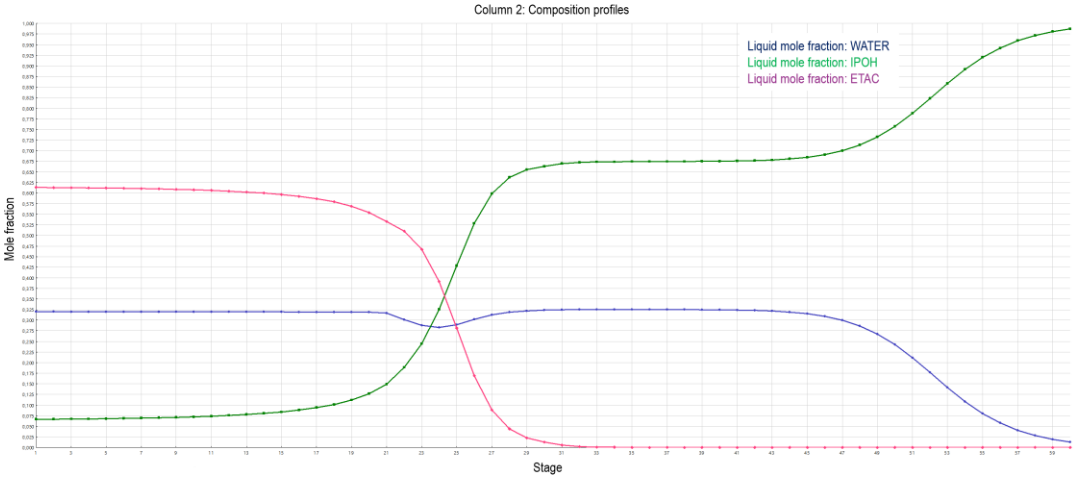


Figure 33: Composition profiles of Column 2

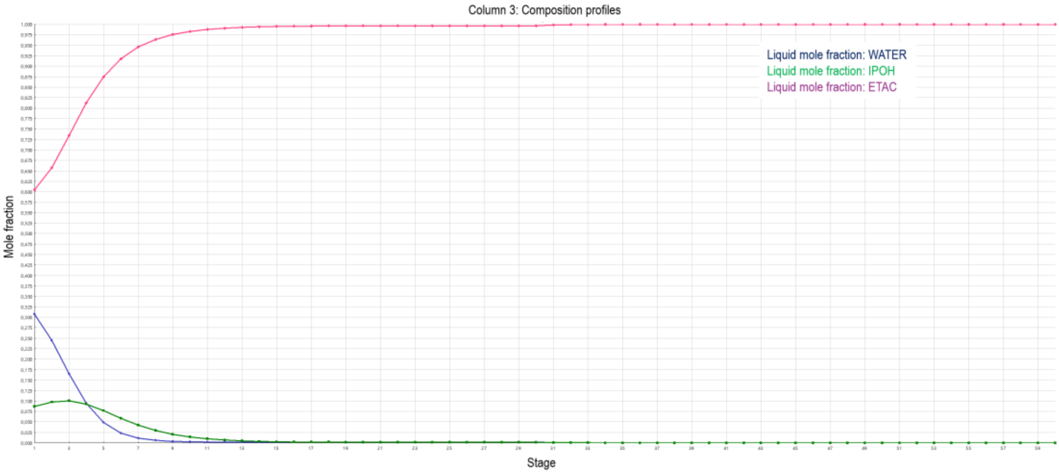


Figure 34: Composition profiles of Column 3

All composition profiles show that the number of stages is high. This is observed because in all the compounds represented for each column, there is an area where the composition remains practically constant. These zones indicate that the number of plates is higher than the required, hence should be reduced.

When the feed stage is at the optimum, in the area where it was constant before, a smooth curve is now observed. Therefore, what should be done is to reduce the number of stages until the optimum plate is found and then vary the initial reflux until it is close to the optimum in each column.

Being close to the column optimum is essential as it is possible to reduce operating and investment cost. Table 19 represents the unoptimized plant and equipment costs.

Table 19: Plant and equipment costs of alternative 3 obtained from Aspen Plus®

Total Capital Cost [USD]	9 117 060	**Electicity [USD/h]**	6.656
Total Operating Cost [USD/Year]	6 441 380	**Cooling water [USD/h]**	36.624
Total Utilities Cost [USD/Year]	4 475 120	**Column 1 Total Cost [USD]**	1 686 900
Equipment Cost [USD]	2 221 300	**Column 2 Total Cost [USD]**	2 838 700
Total Installed Cost[USD]	4 576 600	**Column 3 Total Cost [USD]**	2 130 900

4.5. ALTERNATIVE 4

As discussed in the "Residue Curve Map" section, process 4 and 5 are valid if there is no ternary azeotrope. This implies that the number of columns and the cost of operation would be reduced by simplifying the process.

Alternative 4 (Figure 35) consist of 2 columns, which separates pure ETAC and IPOH, and 1 decanter where it is possible to obtain pure water. Pure ETAC is mixed with crude feed and a ternary mixture (distillate 1) to feed the decanter. Two streams flow out of the decanter, an aqueous one, where practically pure water is obtained, and an organic one provides columns 2.

Pure ETAC is obtained in column 2, where it is splitter in two streams: One goes out the system, and the other is recirculated to the crude feed. The distillate is a ternary mixture that feeds column 1. Pure IPOH is obtained in column 1, and the distillate is recirculated to the decanter.

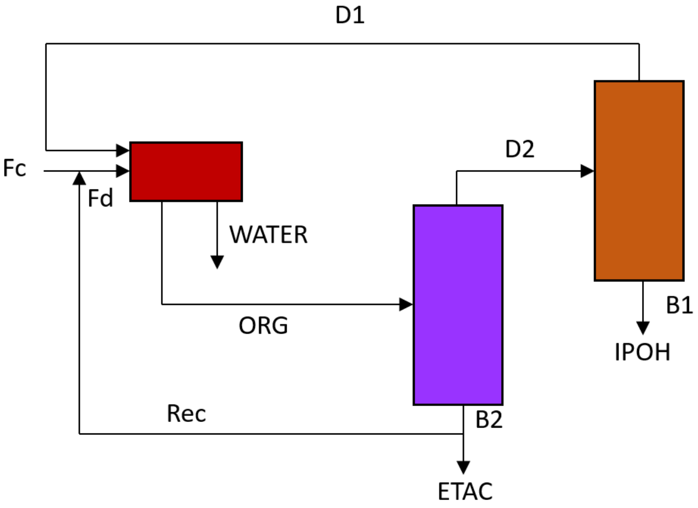


Figure 35: Alternative 4 setup

4.5.1. Residue curve map and DSE equation

In this alternative, no simulations have been carried out because Aspen does not detect the ternary azeotrope in the properties of the mixture, but it does in the simulation. For this reason, there is no DSE value, but it could be expected to be high due to simplicity. Figure 36 shows the residue curve map of Alternative 4.

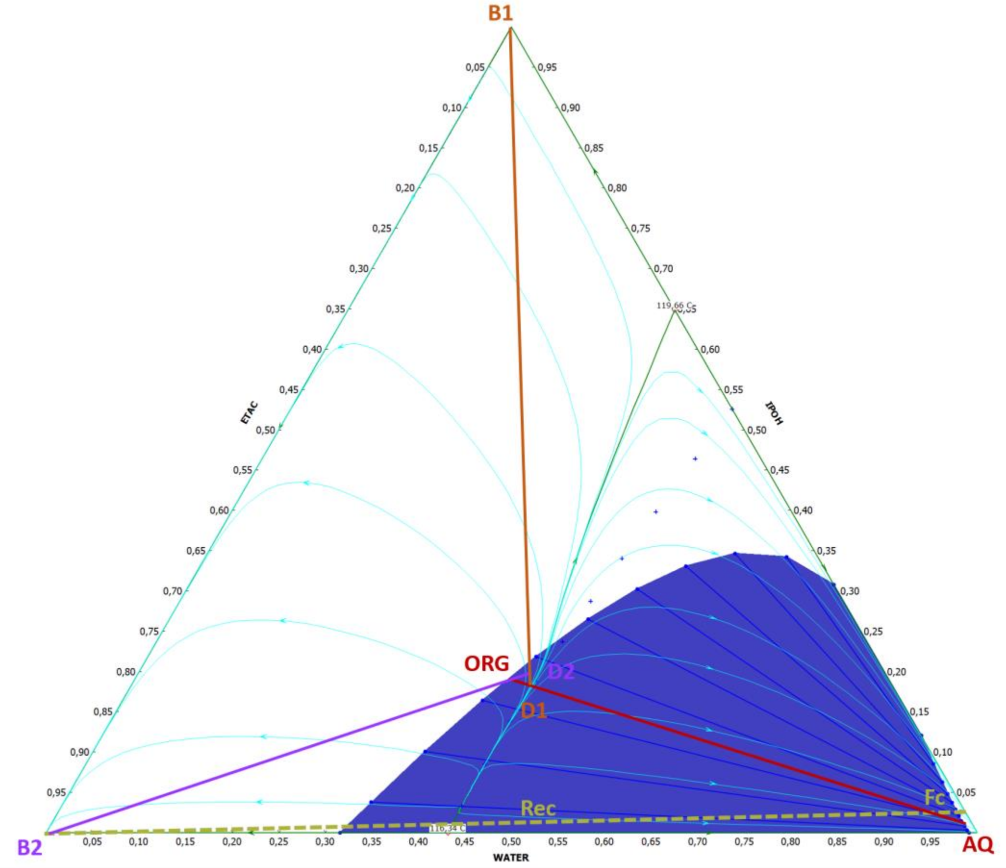


Figure 36: Residue curve map of alternative 4

Equation 19 correspond to the DSE of this alternative. As the process cannot be simulated, it is impossible to know the exact value of the distillation sequence efficiency. If the recirculated flow value is low, the DSE will be high. The term "Rec" does not appear because it moves backwards and forwards with -1 and 1.

$$DSE = \frac{B_1 \cdot \eta_2 + B_2 + Water + D_1 \cdot (\eta_1 \cdot \eta_2 - 1)}{Fc} \quad (19)$$

4.6. ALTERNATIVE 5

Alternative 5 (Figure 37) is in the same case as Alternative 4. This process consists of 2 columns and one decanter.

Crude feed is mixed with a pure ETAC stream comes from bottom of column 2. This mixture feeds column 1, where pure IPOH is obtained from bottom and binary azeotrope (water – ETAC) from distillate is mixed with the distillate of column 2, which has the same composition as distillate 1 (the binary azeotrope).

The decanter separates two phases: an aqueous that is practically pure and an organic one that feeds column.

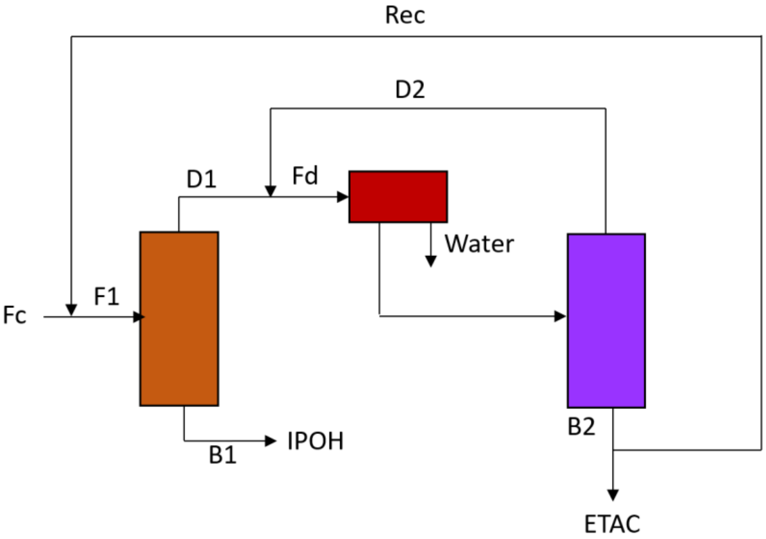


Figure 37: Alternative 5 setup

4.6.1. Residue curve map and DSE equation

The same applies to this alternative as alternative 4. The DSE cannot be calculated because it was not possible to do the simulation. The reason for that is that Aspen detects the ternary azeotrope, and the desired compositions cannot be obtained. Figure 38 shows the residue curve map of alternative 5.

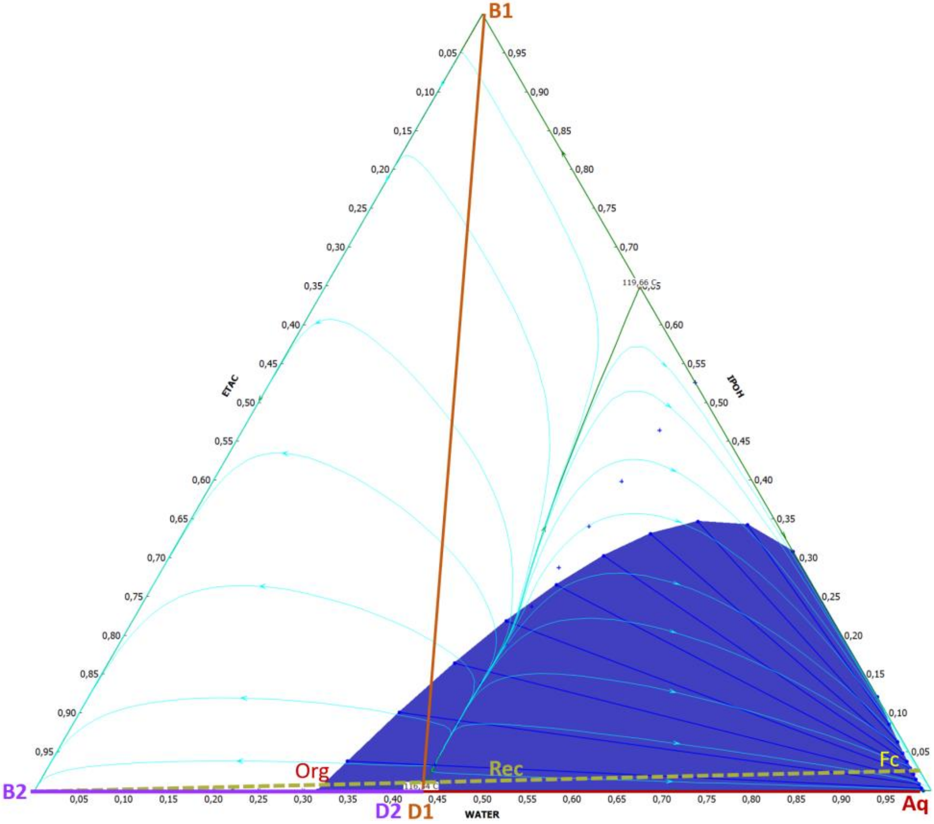


Figure 38: Residue curve map of alternative 5

Equation 20 presents the distillation sequence efficiency of this setup. It is expected to have an elevated efficiency due to it is a simple process where the distillates have the same composition. However, as in alternative 4, since it cannot be calculated, the exact value is unknown.

$$DSE = \frac{B_1 + B_2 \cdot \eta_1 + Wat \cdot \eta_1 + D_2 \cdot (\eta_2 - 1) + Rec \cdot (\eta_1 - 1)}{Fc} \qquad (20)$$

5. ALTERNATIVES COMPARISON

Table 20 is a summary of the efficiencies of each process and the number of equipment used. In this table, there are only the alternatives that could be calculated. Therefore, alternative 3 and 4 do not appear because they would be only feasible fi the ternary azeotrope would not exist.

Table 20: Summary of process characteristics

	Proposal of the literature	Alternative 1	Alternative 2	Alternative 3	Alternative 3 - Variant
DSE	0.954	0.179	-0.292	0.929	0.652
Extractor agent	DMSO	No	No	No	No
Number of columns	4	3	4	3	3
Number of decanters	0	1	1	1	1

As this table shows, there is a significant difference between alternative 2 from the others setup, both in terms of the number of equipment and in terms of efficiency. For this reason, alternative 2 would be the least efficient both economically and in terms of performance.

Focusing on alternative 1 could be a good option as it does not an extractor agent and the number of equipment is reduced.

Alternative 3 and the literature proposal have similar efficiency. Nevertheless, in alternative 3 the number of columns is smaller and more importantly, no extracting agent is used.

This is why it can be considered Alternative 3 as the best option. It is the alternative with the best efficiency, fewer columns, and does not use an extractor agent. This last point is very important due to these compounds will be used in the pharmaceutical industry.

6. CONCLUSIONS

Using residue curve maps and rigorous simulation, it has been verified that it is possible to separate ethyl acetate and isopropanol from water without extracting agents. This is very important because these compounds will be used in the pharmaceutical industry.

All the proposed alternatives are feasible according to the use of the residue curve map and infinite/infinite analysis. However, they could not implement the options at a pressure of 4 atm due to the appearance of the ternary azeotrope. Through adjustment of the thermodynamic model and with new experimental data, they could be implemented.

By studying the DSE it has been found that alternative 3 has practically the same efficiency as the one proposed in the literature, even without the uses a third compound to separate the mixture.

7. FUTURE WORK

As proposals for future work, the following points could be made:

- Optimise the proposed alternatives and carry out an economic and environmental study of each one.
- Propose alternatives that may be more efficient or try to use less equipment to obtain the desired composition.
- Try to implement a process control and automation system.
- To deepen the design of the columns: Choice the optimal column tray, column try dimensions, head type, among other options.
- Following the previous point, do the same for the decanters.
- Try to make an instrumentation and piping diagram of the chosen process.

These are some of the points that could be made in the future, but there are probably more.

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ACRONYMS

ETAC	Ethyl acetate
IPOH	Isopropyl alcohol
DMSO	Dimethyl sulfoxide
EG	Ethylene glycol
DSE	Distillation sequence efficiency
Fc	Crude feed flow rate
RCM	Residue curve map
NRTL	No Random Two Liquids
VLE	Vapour – Liquid equilibrium
η_i	Carnot efficiency of column i
W_i	Mole flow rate of stream i
T_{Bi}	Temperature bottom of stream i
T_{Di}	Temperature distillate of steam i
$x_{(stream)(compound)}$	Molar fraction of the compound
MMB	Macroscopic mass balance

APPENDICES

APPENDIX 1: BALANCES MATHEMATICA CODE

Alternative 1:

```
Quit []

(* Data *)
fc = 100; xfcw = 0.9697; xfcip = 0.0258; xfcetac = 0.0045;
xb1w = 0.99; xb1ip = 0.01;
xb3w = 0.01; xb3ip = 0.99;
xb2etac = 0.99; xb2ip = 0.01; xb2w = 0;
xd3w = 0.321; xd3ip = 0.065; xd3etac = 0.614;
xaqw = 0.9758858; xaqip = 0.011809;
xorgw = 0.25320; xorgip = 0.0713528;
(* degree of freedom *)
xd1w = 0.93;

(* Balances *)
(* Overall balance *)
fc == b1 + etac + b3;
fc*xfcw == b1*xb1w + etac*xb2w + b3*xb3w;
fc*xfcip == b1*xb1ip + etac*xb2ip + b3*xb3ip;
(* decanter *)
fc + d3 + rec + d1 == org + aq;
fc*xfcw + d3*xd3w + rec*xb2w + d1*xd1w == org*xorgw + aq*xaqw;
fc*xfcip + d3*xd3ip + rec*xb2ip + d1*xd1ip == org*xorgip + aq*xaqip;
(* Column 1 *)
aq == d1 + b1;
aq*xaqw == d1*xd1w + b1*xb1w;
aq*xaqip == d1*xd1ip + b1*xb1ip;
```

```
(* Column 2 + split *)
org == d2 + rec + etac;
org*xorgw == d2*xd2w + rec*xb2w + etac*xb2w;
org*xorgip == d2*xd2ip + rec*xb2ip + etac*xb2ip;
(* Column 3 *)
d2 == d3 + b3;
d2*xd2w == d3*xd3w + b3*xb3w;
d2*xd2ip == d3*xd3ip + b3*xb3ip;

(* Resolution of mass balance: 13 unknows, 13 equations, 1 degree of \
freedom *)
Solve [fc == b1 + etac + b3 &&
  fc*xfcw == b1*xb1w + etac*xb2w + b3*xb3w &&
  fc*xfcip == b1*xb1ip + etac*xb2ip + b3*xb3ip &&
  fc + d3 + rec + d1 == org + aq &&
  fc*xfcw + d3*xd3w + rec*xb2w + d1*xd1w == org*xorgw + aq*xaqw &&
  fc*xfcip + d3*xd3ip + rec*xb2ip + d1*xd1ip ==
   org*xorgip + aq*xaqip && org == d2 + rec + etac &&
  org*xorgw == d2*xd2w + rec*xb2w + etac*xb2w &&
  org*xorgip == d2*xd2ip + rec*xb2ip + etac*xb2ip && d2 == d3 + b3 &&
  d2*xd2w == d3*xd3w + b3*xb3w && d2*xd2ip == d3*xd3ip + b3*xb3ip,
 {b1, b3, etac, d1, d2, d3, aq, org, rec, xd1ip, xd2w, xd2ip}]
```

Alternative 2:

```
Quit []

(* Data *)
xorgw = 0.25629; xorgip = 0.07029; xorgetac = 0.67341;
xaqw = 0.97655; xaqip = 0.01131; xaqetac = 0.01213;
xd2w = 0.321; xd2ip = 0.065; xd2etac = 0.614;
xd4w = 0.327; xd4ip = 0.673;
fc = 100; xfcw = 0.9697; xfcip = 0.0258; xfcetac = 0.0045;
xb1w = 0.99; xb1ip = 0.01;
```

```
xb4w = 0.01; xb4ip = 0.99;
xb3etac = 0.99; xb3ip = 0.01;
(* Degree of freedom *)
xb2ip = 0.673;

(* Balances *)
(* Mixer 1*)
fc + d4 == f1;
fc*xfcw + d4*xd4w == f1*xf1w;
fc*xfcip + d4*xd4ip == f1*xf1ip;
(* Column 1 *)
f1 == d1 + b1;
f1*xf1w == d1*xd1w + b1*xb1w;
f1*xf1ip == d1*xd1ip + b1*xb1ip;
(* Mixer 2 *)
d1 + d3 == f2;
d1*xd1w + d3*xd3w == f2*xf2w;
d1*xd1ip + d3*xd3ip == f2*xf2ip;
(* Column 2 *)
f2 == d2 + b2;
f2*xf2w == d2*xd2w + b2*xb2w;
f2*xf2ip == d2*xd2ip + b2*xb2ip;
(* Decanter: 1 lineal *)
d2 == aq + org;
d2*xd2w == aq*xaqw + org*xorgw;
d2*xd2ip == aq*xaqip + org*xorgip;
(* Column 3 *)
org == d3 + b3;
org*xorgw == d3*xd3w + b3*0;
org*xorgip == d3*xd3ip + b3*xb3ip;
```

```
(* Column 4: An equation is a linear combination. Two appear in solver*)
b2 == b4 + d4;
b2*xd2w == b4*xb4w + d4*xd4w;
b2*xd2ip = b4*xb4ip + d4*xd4ip;
(* Boundary conditions *)
xd1w == -1.495*xd1ip^3 + 2.3769*xd1ip^2 - 1.0989*xd1ip +
  0.4474; xd3w == -0.1034*xd3ip + 0.3294;

(* Resolution of mass balances: 20 unknowns, 20 equations, 1 degree of freedom *)
Solve [fc + d4 == f1 && fc*xfcw + d4*xd4w == f1*xf1w &&
  fc*xfcip + d4*xd4ip == f1*xf1ip && f1 == d1 + b1 &&
  f1*xf1w == d1*xd1w + b1*xb1w && f1*xf1ip == d1*xd1ip + b1*xb1ip &&
  d1 + d3 == f2 && d1*xd1w + d3*xd3w == f2*xf2w &&
  d1*xd1ip + d3*xd3ip == f2*xf2ip && f2 == d2 + b2 &&
  f2*xf2w == d2*xd2w + b2*xb2w && f2*xf2ip == d2*xd2ip + b2*xb2ip &&
  d2 == aq + org && d2*xd2w == aq*xaqw + org*xorgw && org == d3 + b3 &&
  org*xorgw == d3*xd3w + b3*0 && org*xorgip == d3*xd3ip + b3*xb3ip &&
  b2 == b4 + d4 && b2*xd2w == b4*xb4w + d4*xd4w &&
  xd1w == -1.495*xd1ip^3 + 2.3769*xd1ip^2 - 1.0989*xd1ip + 0.4474 &&
  xd3w == -0.1034*xd3ip + 0.3294,
 {b1, b2, b3, b4, d1, d2, d3, d4, f1, f2, xd1w, xd1ip, xd3w, xd3ip,
  xb2w, xf1w, xf1ip, xf2w, xf2ip, aq, org}]
```

Alternative 3:

```
Quit []

(* Data *)
fc = 100;
xfcw = 0.97; xfcip = 0.025;
xorgw = 0.2563; xorgip = 0.071;
xaqw = 0.976; xaqip = 0.011;
xd2w = 0.321; xd2ip = 0.065;
xb1w = 0.99; xb1ip = 0.01;
```

```
xb2w = 0.01; xb2ip = 0.99;
xb3etac = 0.99; xb3ip = 0.01; xb3w = 0;

(* Balances *)
(* Overall balance *)
fc == b1 + b2 + b3;
fc*xfcw == b1*xb1w + b2*xb2w + b3*xb3w;
fc*xfcip == b1*xb1ip + b2*xb2ip + b3*xb3ip;
(* MMB Column 1 + boundary condition *)
fc + aq == b1 + d1;
fc*xfcw + aq*xaqw == b1*xb1w + d1*xd1w;
fc*xfcip + aq*xaqip == b1*xb1ip + d1*xd1ip;
xd1ip == 8.5618*xd1w - 2.086;
(* MMB decanter: 1 es lineal *)
d2 == org + aq; d2*xd2w == org*xorgw + aq*xaqw;
d2*xd2ip == org*xorgip + aq*xaqip;
(* MMB mixer: org +d1 *)
d1 + s == f2;
d1*xd1w + s*xorgw == f2*xf2w;
d1*xd1ip + s*xorgip == f2*xf2ip;
(* Split: 2 lineal xf2=xf3=xr *)
org == s + f3;
org*xorgw = s*xorgw + f3*xorgw;
org*xorgip = s*xorgip + f3*xorgip;
(* MMB column 2 *)
f2 + d3 == b2 + d2;
f2*xf2w + d3*xd3w == d2*xd2w + b2*xb2w;
f2*xf2ip + d3*xd3ip == d2*xd2ip + b3*xb2ip;
(*Column 3 + boundary condition *)
f3 == d3 + b3;
f3*xorgw == d3*xd3w + b3*xb3w;
f3*xorgip == d3*xd3ip + b3*xd3ip;
```

```
0.5228 == xd3w/ (1 - xd3w - xd3ip);

(* Resolution of mass balances: 17 unknowns, 17 equations, 0 degree of freedom*)
Solve [fc == b1 + b2 + b3 &&
  fc*xfcw == b1*xb1w + b2*xb2w + b3*xb3w &&
  fc*xfcip == b1*xb1ip + b2*xb2ip + b3*xb3ip && fc + aq == b1 + d1 &&
  fc*xfcw + aq*xaqw == b1*xb1w + d1*xd1w &&
  fc*xfcip + aq*xaqip == b1*xb1ip + d1*xd1ip &&
  xd1ip == 8.5618*xd1w - 2.086 && d2 == org + aq &&
  d2*xd2w == org*xorgw + aq*xaqw && d1 + s == f2 &&
  d1*xd1w + s*xorgw == f2*xf2w &&
  d1*xd1ip + s*xorgip == f2*xf2ip && org == s + f3 && f3 == d3 + b3 &&
  f3*xorgw == d3*xd3w + b3*xb3w &&
  f3*xorgip == d3*xd3ip + b3*xb3ip &&
  0.5228 == xd3w/(1 - xd3w - xd3ip), {b1, b2, b3, aq, org, s, d1, d2,
  d3, f2, f3, xd1w, xd1ip, xf2w, xf2ip, xd3w, xd3ip}]
```

APPENDIX 2: MMB CHANGING DEGREES OF FREEDOM: ALTERNATIVE 1 AND 2

Alternative 1:

Stream	Fc	Org	Aq	D1	B1	D2	B2
Mole flow [kmol/h]	100	87.772	151.331	53.398	97.933	70.796	16.977
	Mole fraction						
Water	0.970	0.253	0.976	0.940	0.990	0.314	0.000
IPOH	0.026	0.071	0.012	0.015	0.010	0.086	0.010
ETAC	0.004	0.675	0.012	0.045	0.000	0.600	0.990

Stream	D3	B3	Rec	ETAC
Mole flow [kmol/h]	69.183	1.612	16.522	0.455
	Mole fraction			
Water	0.321	0.321	0	0
IPOH	0.065	0.065	0.010	0.010
ETAC	0.614	0.614	0.990	0.990

xd1w = 0,95
DSE
0.0848

Alternative 2:

Stream	**Fc**	**F1**	**F2**	**Org**	**Aq**	**D1**	**B1**
Mole flow [kmol/h]	100	234.190	139.556	2.528	0.250	137.535	96.655
	Mole fraction						
Water	0.970	0.601	0.328	0.256	0.977	0.328	0.990
IPOH	0.026	0.397	0.660	0.070	0.011	0.668	0.010
ETAC	0.004	0.002	0.012	0.673	0.012	0.003	0

Stream	**D2**	**B2**	**D3**	**B3**	**D4**	**B4**
Mole flow [kmol/h]	2.777	136.779	2.021	0.507	134.190	2.589
	Mole fraction					
Water	0.321	0.327	0.321	0	0.327	0.010
IPOH	0.065	0.673	0.085	0.010	0.673	0.99
ETAC	0.614	0.000	0.594	0.990	0.000	0

xb2ip = 0,672
Càlcul DSE
-0.386