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Basell Poliolefinas Ibérica SL

Energy Optimization of a Propene-Propane Splitter

Master thesis presented by Carlos José Guindán

to obtain the Master degree in Chemical Engineering
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Carlos José Guindán

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NOMENCLATURE

Acronym

AHPs	
AVG	Average value
BWRS	Benedict-Webb-Rubin-Starling correlations
CPA	Cubic-Plus-Association correlations
CRHPs	Absorption heat pumps
CS	Control surface
CV	Control volume
D	Detected
DCS	Distributed Control System
FP	Fluid package
FSA/B/C	Feed stream A, B or C
GC	Gas chromatography
HECO	Hetero copolymers
HIDiC	Heat integrated distillation column
HK	Heavy key component
HOMO	Homopolymers
HP	Heat pump distillation technology
IP21	Aspen InfoPlus.21® software
LK	Light key component
LYB	LyondellBasell company
MVR	Mechanical vapour recompression
NCG	Non-condensable gases
ND	No detected
OOS	Out of sample
P&ID	Piping and instrumentation diagram
PDH	Propane dehydrogenation plant - BASF SONATRACH Propanchem S.A.
PFD	Process flow diagram
PPII	Polypropylene II – Manufacturing line
PPIII	Polypropylene III – Manufacturing line
PR	Peng Robinson correlations
PRSV	Peng-Robinson Stryjek-Vera correlations
RACO	Random copolymers
SD	Standard deviation

SFS	Simplified feed stream
SRK	Soave Redlich Kwong correlations
TA	PFD top vapour temperature
TAHP	Thermo-acoustic heat pump
TAO	Tarragona Olefins
TB	PFD reflux temperature
TBP	True boiling point temperature
TC	W655 datasheet top vapour temperature
TD	W655 datasheet outlet stream temperature
TQS	Department of Technologies, Quality and Site Services
TVR	Thermal vapour recompression
UNIQUAC	Universal Quasi-chemical
VC	Vapour compression
VF	Vapour fraction
VRC	Compression–resorption heat pumps

Symbols

ΔT	Temperature difference between bubble and dew point
ρ	Liquid density in kg/m3
C_i	Cost of the utility i
E_{MVP}	Murphee’s point efficiency
E_{MVT}	Murphee’s tray efficiency
E_0	Distillation column global efficiency
G	Distilled mass flow rate in kg/h
G_{1L}	Mass flow rate of the liquid phase of stream 1
G_{1V}	Mass flow rate of the vapour phase of stream 1
G_2'	Restricted reflux mass flow rate
$G1$	Main condensers outlet stream mass flow rate
$G2$	Reflux mass flow rate
$G3$	Vent gas flow rate
$G4$	Vent condenser inlet stream flow rate
$G5$	Vent condenser outlet stream flow rate
k_i	Utility i-unit annual price in €/year*kW

M	Reflux drum liquid phase total mass
MAI	Model adjustment index
MSE	Mean Square Error
$MSRE$	Mean square
$MSRE$	Mean Square Relative Error
L_1	Liquid flow leaving tray 1 of column K656.
N_R	Real number of stages
N_T	Theoretical number of stages
$P_{PROPYLENE}$	Propylene selling price in €/kg
Q_i	Heat exchanger duty in KW
RE	Relative error
$RMSE$	Root Mean Square Error
$RMSRE$	Root mean Square Relative Error
VF, t_V	Vapour fraction
VF_D, t_{VD}	Design vapour fraction
VT_0	Volume of liquid at the initial time t0
VT_1	Volume of liquid in the tank after time t1 corresponding to the total duration of the reflux valve closure test.
V_1	Inlet steam current to tray 1 of column K656.
$\hat{Y}$	Predicted values
Y_D	Observed values
t_{OP}	Annual operating time in h/year
$w_{PROPYLENE}$	Distilled propylene mass fraction (%w/w)
y	Outlet vapour composition in a specific point in the tray
y^*	Hypothetical composition of a vapor phase in equilibrium with a liquid phase with the same composition of the real outlet stream in the considered point
y_n	Outlet vapour composition coming from stage n
${y_n}^*$	Hypothetical composition of a vapor phase in equilibrium with a liquid phase with the same composition of the real outlet stream
y_{n+1}	Inlet vapour composition coming from the stage n+1

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SUMMARY

This project boards the energy optimization of the propylene/propane distillation, with a cost-benefit basis. The work placement was at LyondellBasell's [redacted] plant, to optimize the propylene recovery section of the Polypropylene II (PPII) production line.

The project first phase was to collect and study the basic design engineering. The design criteria and the operating conditions of the section equipment were obtained. The feed composed of lighter and heavier hydrocarbons than the propane/propylene system led to the design of a column of [redacted] plates divided into two shorter columns [redacted]). A [redacted] remaining propylene in the vapour phase, increasing thus the mass recovery. The [redacted]. In the design, [redacted] kg/h of carrier gas composed by [redacted] %w/w of propylene and [redacted] %w/w of propane are fed in the column to obtain [redacted] kg/h of product with a [redacted] %w/w of propylene and [redacted] %w/w of propane, operating at a reflux ratio of [redacted]. A study of the state of the art of distillation optimization for the system under study was also carried out.

In the next phase, a theoretical model was built. This simplified the study of the system behaviour to determine optimal operating conditions. The modelling was carried out in Aspen HYSYS® for It great performance to simulate distillation columns. A base model was created, and its performance was analysed by predicting the design specifications. The influence of the type of components included in the feeding modelling was evaluated, as well as the fluid package that generated the best fit model/design. The best feed stream alternative was the one excluding heavy hydrocarbons. Peng Robinson was the fluid package that provided the best data fit. This was evaluated with an index based on the mean square relative error comparing model design and predictions. This index value of 0.0602 indicates an average relative error of 6.02%.

In the phase three the distillation current operating conditions were obtained. Some historical data was obtained from the Distributed Control System (DCS) and local measurement instruments. The most relevant streams composition was determined through chromatography analysis on extracted samples. Other intermediate variables were established with empirical industrial scale tests using the installed instrumentation. To conclude, the equilibrium of the liquid feed is simulated to estimate the compositions feed's vapour phase, thus being completely defined: [redacted] respectively was fed to the column. Operating at a reflux ratio of [redacted] kg/h of product is obtained with [redacted] and [redacted] %w/w of propylene and propane content.

With the data of the real process, the model was calibrated and optimized using the Aspen HYSYS® Optimizer module introducing the actual feed stream data. Operating at an optimal reflux ratio of [redacted], [redacted] kg/h of distillate is produced with [redacted] %w/w propylene and [redacted] %w/w propane. This operation requires [redacted] m3/h of cooling water, [redacted] kg/h of low-pressure steam and [redacted] kg/h of propylene for refrigeration. The utilities maximum supply capacity was analysed, and the utilities flow rates needed for optimal operation can easily be provided nowadays.

1. INTRODUCTION

Global energy crisis has a major impact in every activity carried out these days. In the industry, this introduces limitations to grow and be competitive in each market. Therefore, despite the type of industry, key challenges are faced to ensure and guarantee future supply of energy.

Plastics industry besides develops solutions to tackle the waste environment impact also face the challenge that the energy crisis puts on the table. In this context, many attempts are taking place to reduce energy consumption, improve processes efficiency or develop new emerging technologies.

1.1. Global Energy Crisis

Over the years, humankind history is characterized by improvement. Humans were always seeking to transcend the limits of the possible by developing new materials and technologies to improve quality of life. Civilization progress brought positive improvements in our lives like new goods and services. At the same time this created important issues and challenges to address related with this comfort. The explosive consumption of available natural resources and the environmental impact is one of the major concerns linked to the progress.

Our modern life is designed to highly depend on energy. In fact, coming from different sources along the years human being has always needed it. Unfortunately, population growth, industrialization, and urbanization (among other factors) linked to increasing consumption of traditional non-renewable energy sources are causing climate change.

In the last decades, several strategies to reduce climate change were proposed. These are based in developing and introduce new technologies, increase the efficiency in the use of energy and encourage the industry to create environmental-friendly substitute process.

One important recent regulation in this direction is the CO2 tax implemented by the European Union. This is intended to limit the consumption while reducing the demand for fossil fuels to finally decrease their output of greenhouse gases and carbon dioxide. This tax has a direct impact in the operating cost. Therefore, optimize the energy consumption is a first step to achieve a minimum operating cost while a transition (if it's possible) from a traditional process to a more sustainable alternative is accomplished.

1.2. Propylene and Polypropylene (PP)

Propylene is one of the most important petrochemicals produced globally today. In 2015, the annual production capacity was estimated around 114 million tons (Chang Chu, 2017), taking the second position behind ethylene among organic chemicals. However, the global demand for propylene has been projected to grow at a rate of 5% annually, faster than that of ethylene (Chang Chu, 2017).

As a raw material, propylene has maintained a remarkable growth in the worldwide market. This is mainly linked to the raising demand for polypropylene taking more than two-thirds of all the produced propylene around the globe. Quality requirements for this purpose is a polymer-grade propylene with a purity higher of 99.5% by weight.

Polypropylene is a promising substitute for polyethylene due to its similar mechanical characteristics and low density. A wide catalogue of products is possible using several novel catalysts and manufacturing methods where Ziegler-Natta catalyst types continue to lead the market.

Plastic industry dates from 1900's but it wasn't after 1950's that the cost of manufacturing decreased to levels low enough to trigger the mass production. Plastic products are strongly present in our daily life reaching vast quantities of products manufactured. However, this also generates equivalent amounts of waste that if it's not properly managed can lead to environmental pollution. Despite this, plastic demand remains as a very important commodity that is projected to keep growing in the future.

In term of companies leading the market, some of the key players within the plastics industry include ExxonMobil, Sinopec, and ***LyondellBasell*** (statista.com).

1.3. [redacted]® Manufacturing process

Tarragona East Site produce polypropylene using the [redacted] developed by Lummus Technology. Characterized by an [redacted] catalysts in [redacted].

The overall process can be reduced into several sections:

[redacted]

Inside the reactor the polymer produced is a powder mixed with the non-reacted gas phase. Next, to separate solid and gas (called *Carrier gas*) phases the reactor discharge is sent to a cyclone at atmospheric pressure. Any unreacted monomer is compressed and either recycled or returned to the upstream olefins' unit for recovery. The final degassing of the polymer is done in a purge vessel by flushing with nitrogen. The purged off gas is sent to a recovery system. Finally, the powder is converted into pellets that incorporate a range of well-dispersed additives.

This project focus on the recovery section defined in the Figure 1.1. Distillation is fed by the reactor outlet stream to separate the non-reacted propylene from other gaseous components to send it back as a recycle stream to the raw material purification and feeding section. The propylene recovery is performed by a C-3 Splitter (distillation column), divided into two smaller columns, with a fully automated operation.

[redacted]

Figure 1.1. Carrier Gas Distillation flowsheet

1.4. Distillation

1.4.1. Fundamentals

Distillation is one of the most widely used separation process in the chemical industry (Choudari A, 2012). A vapour phase in contact with a liquid phase where simultaneously, mass is transferred from the liquid to the vapour by evaporation and from the vapor to the liquid by condensation. This produces a redistribution of components between both phases based on relative volatility. In general, liquid and vapor contain the same components although in different amounts. The liquid is at its bubble point temperature in equilibrium with vapor at its dew point. The overall effect is a concentration increase in the more volatile components in the vapor phase leaving by top and the less volatile in the liquid in bottoms. As evaporation and condensation involve each components latent heats of vaporization, distillation is a unit operation that requires large amounts of energy.

Distillation is widely used to separate liquid mixtures into almost pure components. High pressure can be used to decrease the volume and/or increase the temperature to facilitate condensation; in other cases, it will be necessary to decrease the pressure to set the boiling point below the thermal decomposition temperature.

To bring the two phases into contact the distillation can be carried out in either trays or a packed bed column. In general, tray alternative is most frequent where each tray represents an equilibrium stage. In real life the equilibrium is not reached, and this is the reason why efficiency concept is introduced. Tray diameter depends first on the volume of the gas phase that rises through the tower. It is undesirable to have a high velocity of the gas through the plates due to a higher pressure drop for this phase. Higher pressure drop may require a longer distance between the plates to generate the needed hydraulic head to produce the downward flow of the liquid from plate to plate.

The feed must be introduced in the column in a specific tray. The best location for this stream input is the stage where the inner flowing phases has similar composition and properties to the feed. This will avoid diluting the feed or the inner streams reducing the mass transfer in that tray. In general, no identical stream is found when analysing the streams flowing between stages. Therefore, the stream with composition and properties most similar to the feed is where this stream is added.

Feed location divides the column into two sections. Top of the column (vapour outlet) is called the enrichment or rectification section because the vapor phase is enriched in the more volatile component. Bottoms (liquid outlet) is known as the stripping section because the most volatile component is stripped out from liquid phase.

1.4.2. Design and operation variables

Distillation is a complex unit operation and as such there are many important variables associated with its operation. Among all these, for a feed with a certain composition and flow rate, at a fixed pressure, there are four that must be considered when designing and operating distillation columns.

A *high purity of the product* is required for no additional purification operations. Furthermore, when obtaining products for sale the price increases for higher purity.

According to general heuristics the cost of raw material represents around the 30% of the total cost of a process. Therefore, it is required that the *recovery percentage in the product stream* must be as high as possible.

In terms of capital cost, the greater the *number of stages* required the greater the investment needed in the separation system. At the same time considering the mass transfer, the higher the equilibrium stages the higher the purity of the distilled product.

Considering the operating cost and given the high amounts of energy associated with the column operation it is required that the separation process is performed with the lowest possible *energy consumption*.

Last, and one of the most important design variables is the *reflux ratio (R)*. Defined as the portion of condensed fluid at the top of the column that returns to the column improving enrichment and stripping of the feed stream. Boundaries of this variable are two: minimum and total reflux. The minimum reflux is that value at which the number of stages required to perform a given separation is infinite. Total Reflux is obtained when top and bottoms products are not removed from the column (infinite reflux ratio) and no feed is introduced in the column. For these operating conditions the number of theoretical plates required is a minimum. If the reflux ratio is reduced (by taking off product), the number of plates required increases.

These last two definitions of minimum reflux and minimum number of stages at full reflux are useful to estimate how difficult a separation could be, for setting the operating reflux ratio, and for analysing column performance.

1.4.3. Efficiency

The number of theoretical plates depends on the complexity of the separation to perform and is only determined by mass and energy balances and equilibrium considerations. In a real column, the phases leaving a stage are not in equilibrium, mainly due to insufficient contact time or inadequate dispersion of both phases. The composition change for each phase in a real stage is usually less than that possible in an equilibrium stage.

The operation of a real stage depends on many physical variables. The stage must bring two phases into intimate contact and then separate them. Likewise, for a more intimate and longer contact of phases the outlet streams will be closer to the equilibrium assumption. Among the variables involved are the interfacial area between the phases, the transfer rate (per unit area) of the components between the phases, and the residence time of the phases within the stage.

The efficiency of the stage or plate and therefore the number of actual plates is determined by the system (composition and properties), mechanical design and geometry (plate type and dimensions) and the operating and flow conditions (rates of throughput). To relate the number of equilibrium (theoretical) stages calculated in the design phase and the number of actual stages to build, the stage efficiency is defined. There are several efficiencies to express the impossibility of reaching equilibrium in a real stage, but the most used from a practical point of view is the Global Efficiency (E).

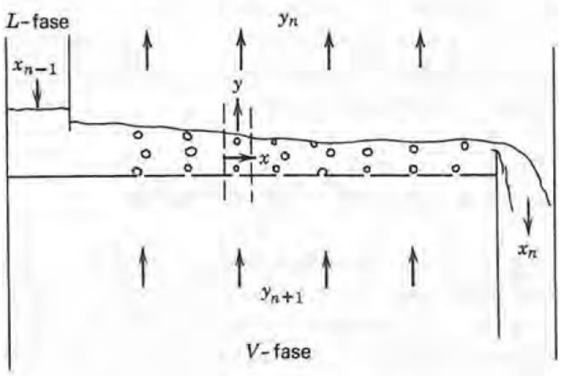


Figure 1.2. Phases contact. Single stage. (Foust, 2006)

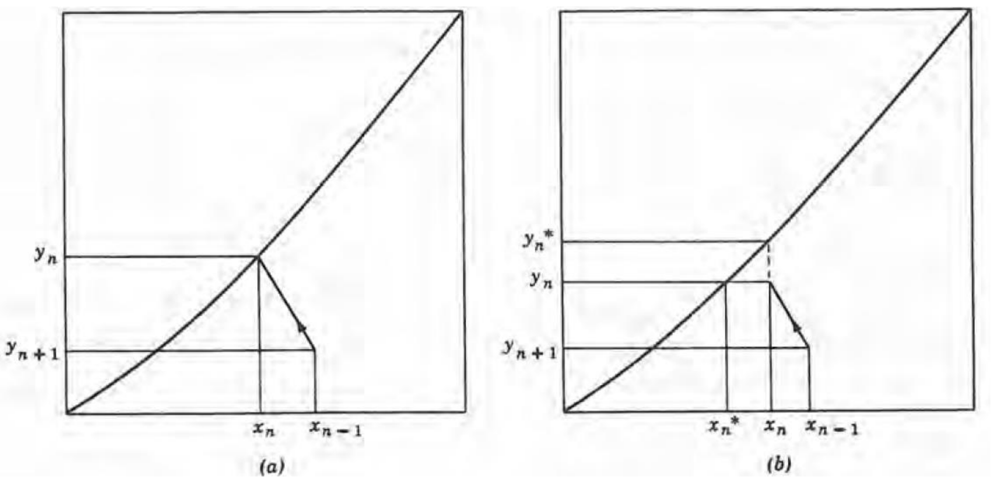


Figure 1.3. Operation line for a single stage. (a) Equilibrium stage. (b) Real stage. (Foust, 2006)

The Global Efficiency consider the whole column (Equation (1.1). Instead, Murphee’s Efficiency (point and tray) considers a specific stage. Both can be defined referring to the liquid phase or the vapor phase (Equations (1.2 and (1.3).

$$E_0 = \frac{Theoretical\ stages}{Real\ stages} = \frac{N_T}{N_R} \tag{1.1}$$

$$E_{MVT} = \frac{y_n - y_{n+1}}{{y_n}^* - y_{n+1}} \tag{1.2}$$

$$E_{MVP} = \frac{y - y_{n+1}}{y^* - y_{n+1}} \tag{1.3}$$

1.4.4. Optimal reflux rate

The optimal reflux ratio is defined by an operation at a minimum total cost. As mentioned previously, for the minimum reflux ratio the column requires an infinite number of stages or trays; consequently, capital cost is infinite but the operating costs (reboiler and condenser duty, and reflux pump power) are minimal. Increasing R, the number of trays decreases rapidly, but the column diameter increases due to the higher mass of liquid and vapor phases flowing through the column. The condenser, reflux pump, and reboiler must also be larger. Therefore, the fixed costs decrease to a minimum and grow again to infinity in the case of operating at full reflux. Here, the heating and cooling requirements increase almost directly with the reflux ratio, as shown. Therefore, the total cost, which is the sum of operating cost and fixed cost, must reach a minimum in the optimal reflux ratio. Often, but not always, this will happen at a reflux ratio close to the minimum value of between 1.2 to 1.5 times the minimum reflux ratio.

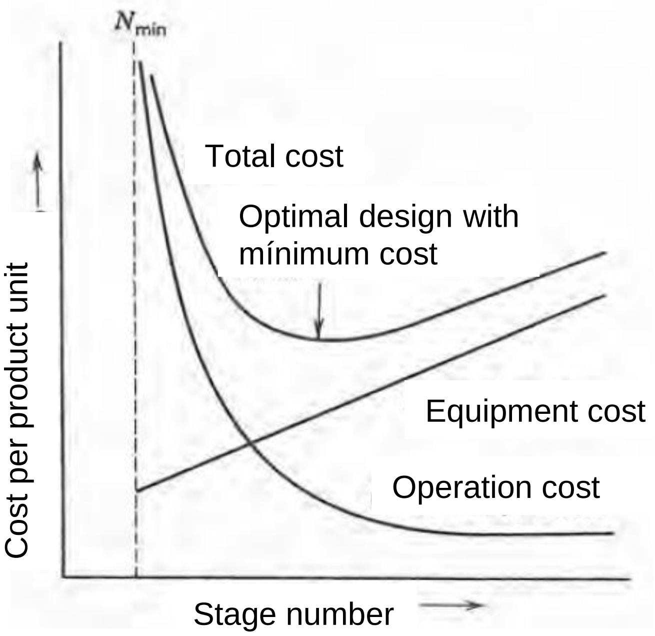


Figure 1.4. Optimal reflux rate in a distillation column

1.4.5. Advanced technologies

To reduce the specific energy consumption several technologies were developed redesigning the traditional arrangement for distillation process. Among several techniques vapor recompression is a widely spread technology to reduce the consumption of energy and can also be found these days in others unit operations (e.g., evaporators) (Kiss et al, 2013). Some of these technologies are:

- ✓ **Heat pump (HP)** systems can be used to upgrade the low-quality energy in the condenser to drive the reboiler of the column.
- ✓ **The vapour compression (VC)**, **thermal and mechanical vapor recompression (TVR and MVR)** technologies are used to upgrade the heat by compressing the vapor distillate or a working fluid.
- ✓ **Compression–resorption heat pumps (CRHPs) and absorption heat pumps (AHPs)** increase the energy efficiency by means of absorption equilibrium.
- ✓ Due to the higher temperature lifts, the **thermo-acoustic heat pump (TAHP)** has a broader applicability range.
- ✓ The internally **heat integrated distillation column (HIDiC)** enhances both the heat and mass transfer, especially when structured packing is used also for exchanging heat.
- ✓ Each and every energy efficient distillation technology yields its maximum savings only at given specific conditions.

2. SCOPE OF THE PROJECT AND SPECIFIC OBJECTIVES

Distillation is one of the most extensively used operations in chemical processes industries with a highly energy consumption associated (Choudhari et al, 2012). The low efficiency makes the distillation process a very energy consuming technology which is responsible for 3% of the world’s energy consumption and 10% of industrial energy consumption (Kazemi et al, 2016), and can generate more than the 50% of the plant operating cost (Kiss et al, 2013).

In the current international scenario of energy crisis during the last years the need to improve the energy consumption has become an important issue. For the case of unit operations as distillation will help not only reduce operating costs but also redistribute the limited amount of energy available.

This project intends to improve the energy consumption in the distillation process (C3-Splitter) in the Polypropylene II (PPII) manufacturing line on a cost-benefit basis. Therefore, the Master’s Final Project was focused to fulfil the following main objectives:

- ✓ To compile all the design basic engineering information needed for modelling the distillation process.
- ✓ To build a model supported on appropriate and well-founded tools that allows to study and predict the system behaviour in the real plant under specific changes in the operating conditions.
- ✓ To determine the real plant current operating conditions
- ✓ To compare real plant process data with the design operating conditions to identify deviations from the original designed operation.
- ✓ To analyse the effect of the key variables and/or parameters to operate the distillation process considering those that are feasible to modify.
- ✓ To propose actions to improve the plant operation reducing energy consumption and operating cost and increasing product quality.

3. STUDENT'S ROLE IN COMPANY

3.1. The company: LyondellBasell

LyondellBasell (LYB) is one of the largest plastics, chemicals, and refining companies in the world. Manufactures materials and products that are key to advancing solutions to modern challenges. These can be grouped into five key product areas:

- ✓ *Chemicals*. Basic chemicals for the chemical industry which are the building blocks for numerous products used in everyday life.
- ✓ *Polymers*. Versatile plastic resins used to produce a variety of products like packaging, automotive parts, and others.
- ✓ *Advanced polymers*. This includes a diverse product portfolio of compounds and advanced polymers including automotive parts, differentiated packaging, specialty pipe, electronics, consumer appliances and construction materials.
- ✓ *Fuels*. Focused mainly in refining heavy, high-sulphur crude oil into refined products including gasoline and distillates.
- ✓ *Technologies*. Licensor of state-of-the-art manufacturing and process technologies use by chemical and plastics companies in the whole world.

These products and solutions enhance food safety through lightweight and flexible packaging, protect the purity of water supplies through stronger and more versatile pipes, improves the safety, comfort, and fuel of means of transport, and ensure the safe and effective functionality in electronics and appliances. LyondellBasell's products can be found in more than 100 countries (Figure 3.1) as the world's largest producer of polymer compounds and the largest licensor of polyolefin technologies.

The potential of being one of the world's largest producers of plastics and chemicals with products used by millions of people every day also implies responsibility. That's why LYB is working to deliver meaningful progress to address some of the world's most pressing challenges such as helping end plastic waste in the environment, reducing climate change and contributing to a thriving society for employees and the communities where the company operate and the people that depend on their products.

In Spain, LYB is present in the autonomous region of Catalonia and Comunitat Valenciana. Tarragona Site West and Tarragona Site East as a part of one single site (LYB TAO) are focused on producing polypropylene resins and advanced polyolefin products. The polypropylene resins are used to manufacture many of modern life products: housewares, building materials and packaging materials. The polyolefin products are used for automotive parts, electrical appliances, and pipe and sheet applications. The work placement was in Tarragona Site East which is also called [redacted]® due to the technology used for polymerisation reaction.

The plant has two main lines of manufacturing called Polypropylene II (PPII) and Polypropylene III (PPIII) to produce three family of products Homopolymers (HOMO), Hetero copolymers (HECO) & Random copolymers (RACO).

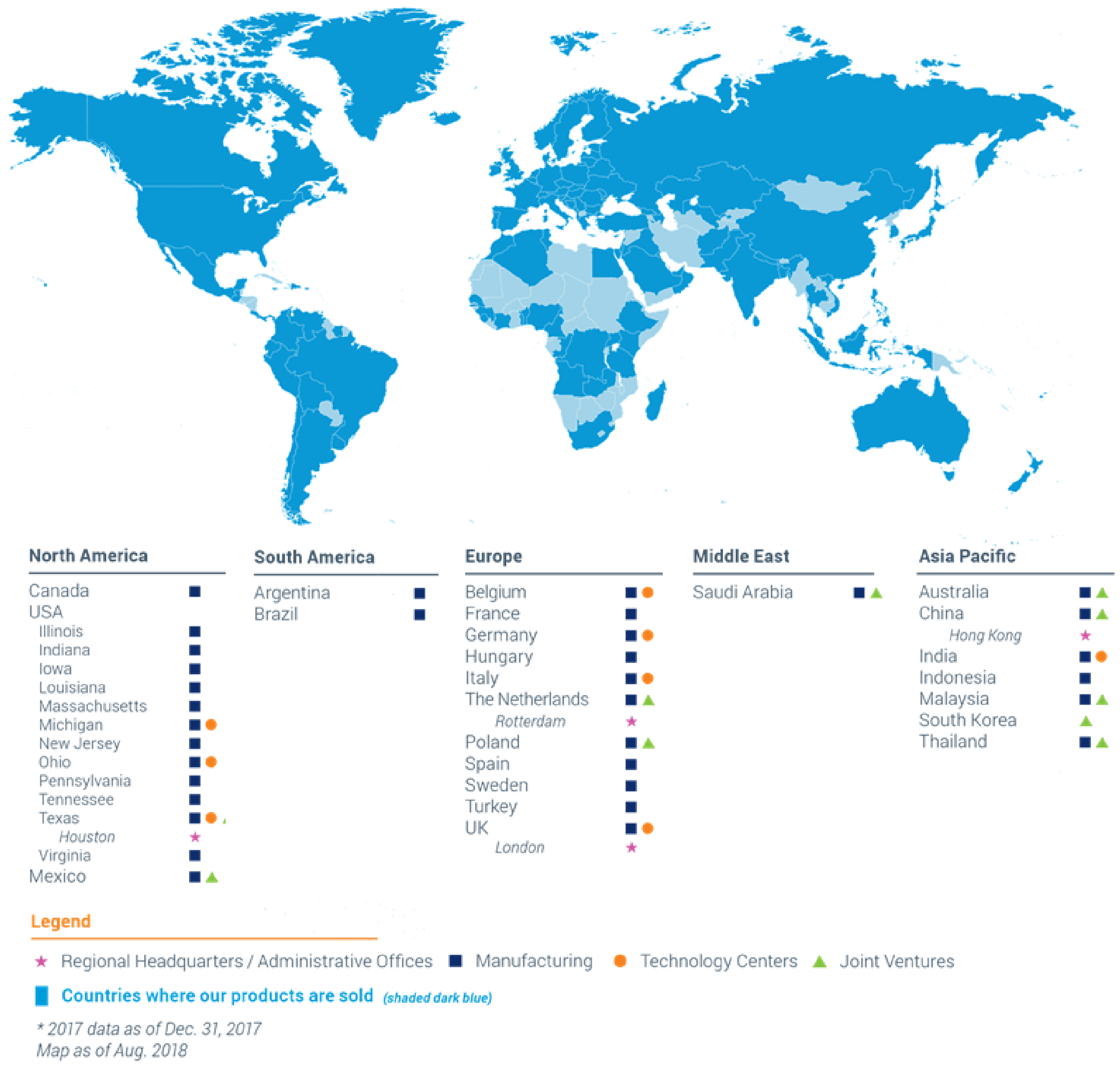


Figure 3.1 - LyondellBasell around the world (adapted from: https://www.lyondellbasell.com)

3.2. <u>Work Placement Activities</u>

Several activities were carried out along the work placement period. Most of them focused on obtaining all the relevant information to perform the Master Final Project. Other, related to provide an overall insight about the role of a Process Engineer in the company.

3.2.1. Internal organization

The work placement position was as Process Engineer Intern and was conceived inside the Department of Technologies, Quality and Site Services (TQS). At the beginning, the tasks related to the work placement were distributed along 18 weeks with a review meeting every Friday. The team for the meetings was mainly composed by:

- ✓ Irene Martos – Process Engineer TQS
- ✓ Marc Viñas Vilaró – Process Engineer TQS – Company Supervisor

- ✓ Victor Alfaro – TQS Manager
- ✓ David Serral – Operation Manager

The objectives of regular meetings were:

- ✓ To check the progress of the tasks
- ✓ To provide support and contribute as a team with different point of views, knowledge and background to propose solutions to raised problems.
- ✓ To define action plans for new tasks defining these inside the boundaries imposed by the scope of the project.
- ✓ To train and improve oral presentation skills.

The company provided access to all the information related to the PPII plant, the software and hardware needed to perform all the scheduled work plan.

3.2.2. Overall activities

From a general point of view, the task executed were the following:

✓ ***To study the state of art of the distillation optimization**.* A screening and research about current strategies to optimize the distillation operations were performed. This provided an initial view of the current situation in that field.

✓ ***To study all the available information provided by the company (P&IDs, set points, operations manuals, etc.).*** All the original design operating conditions and features were defined from the company Basic Engineering Design Reports and Data Sheets. The plant current operating conditions were stablished combining the data showed in the Control Room, the onsite instrumentation, lab tests and simulation assisted estimations. This allowed a further comparison between both steady states and conclude about future actions for improvements.

✓ ***To perform the system modelling and simulation and create the Base Case**.* The information compiled from the Basic Engineering Design was included in the model. This step is assisted by a simulation software creating the C3-Splitter Initial Base Case.

Later, calibration of the initial base case is made loading the current operating conditions in the model. This adjusted model constitutes the Base Case.

Some challenges appeared to converge the flowsheet. Technical support in this field was obtained from URV professors and professionals from LYB to develop new strategies and overcome the obstacle. From the URV, several meetings mostly face to face were carried out with the following professionals:

- **Josep Font Capafons** – PhD in Chemical Sciences – Process Intensification, Simulation and Environmental Technology. 14/11/2022.
- **Carles Torras Font** – PhD in Chemical and Process Engineering – Separation Operations Design. 21/11/2022.
- **Carlos Pozo Fernández** – PhD in Chemical Engineering – Process Engineering Fundamentals. 22/11/2022.

- **Fèlix Lluís Llovell Ferret** – PhD on Chemical Physics - Simulation and Analysis of Chemical Processes, Thermodynamics, and others. 24/11/2022.
- **Carmen Maria Torres Costa** – PhD in Chemical Engineering – Environmental Analysis and Management and Computer Aided Process Engineering. 24/11/2022.

From the company the support was provided by **Rob Fox**, Director of Olefins Process Engineering located in Houston, Texas. Topics discussed were about modelling and simulation and collecting the real plant process data to finally calibrate the model. The meeting was via Microsoft Teams and assisted Marc Viñas Vilaró and Irene Martos from LYB TAO.

After trying new strategies, the flowsheet finally converges.

✓ ***To analyse the effect of reflux rate in the utilities consumption***. From a theoretical approach using the model, the system behaviour for several values of the reflux rate were performed evaluating the impact in the distillation energy consumption and product quality.

After the theoretical evaluation, an industrial test varying the C3 Splitter reflux rate were made next to test the model predictions focusing on the energy consumption and product quality.

With the help of the simulation software the optimization with a cost-benefit basis is executed.

✓ ***To perform a sensitivity analysis***. The optimization procedure is based in the utilities price. To evaluate the optimum estimated a sensitivity analysis of both values is made.

✓ ***To define the Improved Case best operating conditions***. Last, the best operating conditions for minimize the utilities cost are found and proposed.

✓ ***Additional tasks: The role of a Process Engineer***

❖ Process safety.

Several field walkthroughs were performed with colleagues of other areas in LYB to assess the state of the facilities divided by sections. This was made to detect improvement opportunities or risks associated with the operation and maintenance of the plant.

Understanding the fugitive emissions challenge in a hydrocarbons process. Then, provide help to check and modify drawings of the location of the fugitive emissions points in the production lines.

4. METHODOLOGY

4.1. Process Modelling & Simulation

The modelling and simulation procedure is based in three basic steps introduced in Figure 4.1. All the tasks related to building the model along the project were based on this scheme.

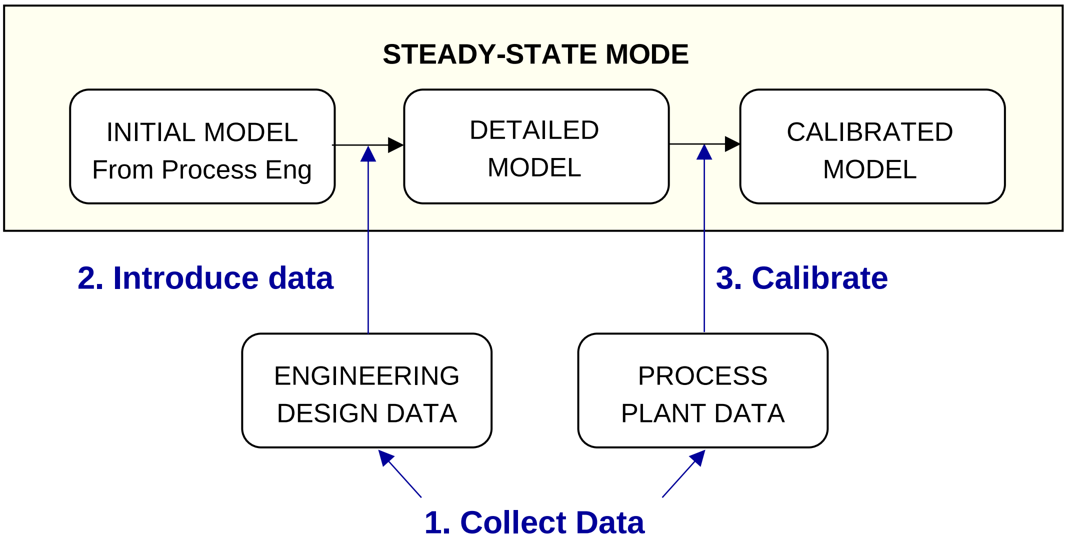


Figure 4.1 – Basic steps to build HYSYS Steady-State models (adapted from Aspentech® library).

4.1.1. Data collection

The sources of information were basically all the engineering data available in the company. For the design data these were: Process Flow Diagrams (PFD), Basic Engineering Report, equipment's data sheets, P&ID's, etc (Appendix A1).

Some discrepancies between the engineering basic data were found. In those cases where a variable or parameter showed different values for each source consulted, the data with the most recent revision date among all of them was taken as valid.

Several field walkthroughs were made as a complement of the basic engineering study combining shorts interviews with production department personnel. The company gave full access to the plant fulfilling the safety requirements. Learning and getting familiarized with the whole manufacturing process allow to understand the operation of distillation columns. Both upstream and downstream operations provide a visualization of boundaries for further modifications.

4.1.2. Model simulation

Distillation modelling is performed with computer aided process simulation software. The two options analysed were Aspen Plus® and Aspen HYSYS® in version v.12. The comparison led to conclude that HYSYS® introduce additional tools and features such as modify the types of equipment and configuration (tower, column, or adding others) of the distillation module inside the Sub Flowsheet Environment called Column Build

Environment. Other advantage of HYSYS is the flexibility of the Dynamics Module suggested for further process control studies.

4.1.3. Model base assumptions

To simplify the model building process, three main assumptions were defined and further analysed about the impact in the results predicted by the model.

a) *Feed components*. Include or not non-condensable gases (NCG) and heavy hydrocarbons compounds.
b) *Main tower configuration*. In terms of mass and energy transfer, splitting the column should not make any difference, but focusing on the simulation software modelling one main column is simpler to simulate than split it into two shorter columns.
c) *Flowsheet*. Similarly, including pumps, valves and others equipment's at this point would not affect the results predicted by the separation process.

4.1.4. Model building procedure

The model was built in the steady state mode following the Aspen HYSYS usual procedure:

- ✓ Load the list of components
- ✓ Select a proper fluid package according to the nature of the system and the unit operations involved
- ✓ Create the flowsheet defining and connecting all the equipment
- ✓ Define inlets
- ✓ Define the operating conditions of all the operations
- ✓ Run the software and generate results

This process was boarded in two steps. In the first step, one basic model was created, simulated and evaluated. Considerations made were: one standard column with a simplified feed stream (SFS) and using Soave Redlich Kwong as a fluid package suggested in the literature (Choudari et al, 2012). Simplified feed stream does not include C4 or heavier hydrocarbons and NCG, this stream only included propylene, ethylene, and propane. In the second step, the vent column with the vent condenser (Figure 1.1) were added to the first step model using previous results to facilitate the flowsheet convergence. An initial base case is built and saved.

Basic information about the fluid package and minimum reflux rate was not found in the basic engineering data. Therefore, from this initial base case and considering the assumptions made and the need to evaluate the best fitting fluid package, several cases of study were defined. For the main tower configuration assumption, the column was modelled considering a single column with a total number of stages equal to the sum of the stages of the two smaller columns designed. For the flowsheet assumption, just the main elements were included: columns, reboiler and condensers. Considering the feed components assumption and the fluid packages as variables to evaluate, three cases of study were finally tested (Table 4.1).

The fluid packages alternatives were chosen using the model assistant provided by HYSYS (Figure 4.2) and the literature available related to this system under study.

Using the Aspen® Plus shortcut distillation module, reflux ratio boundaries and minimum number of stages are estimated.

Table 4.1 - Cases of study

		CASE		
	Variable	A	B	C
1	Feed stream components	Ethylene Propane Propylene	Ethylene Propane Propylene Hrydrogen Nitrogen	Methane Ethane Ethylene Propane Propylene Hydrogen Nitrogen n-Butylene
2	Fluid package	PR SRK PRSV PRSV	PR SRK PRSV PRSV	PR SRK PRSV PRSV

References:

- PR = Peng Robinson
- SRK = Soave-Redlich-Kwong
- BWRS = Benedict-Webb-Rubin-Starling
- PRSV = Peng-Robinson Stryjek-Vera

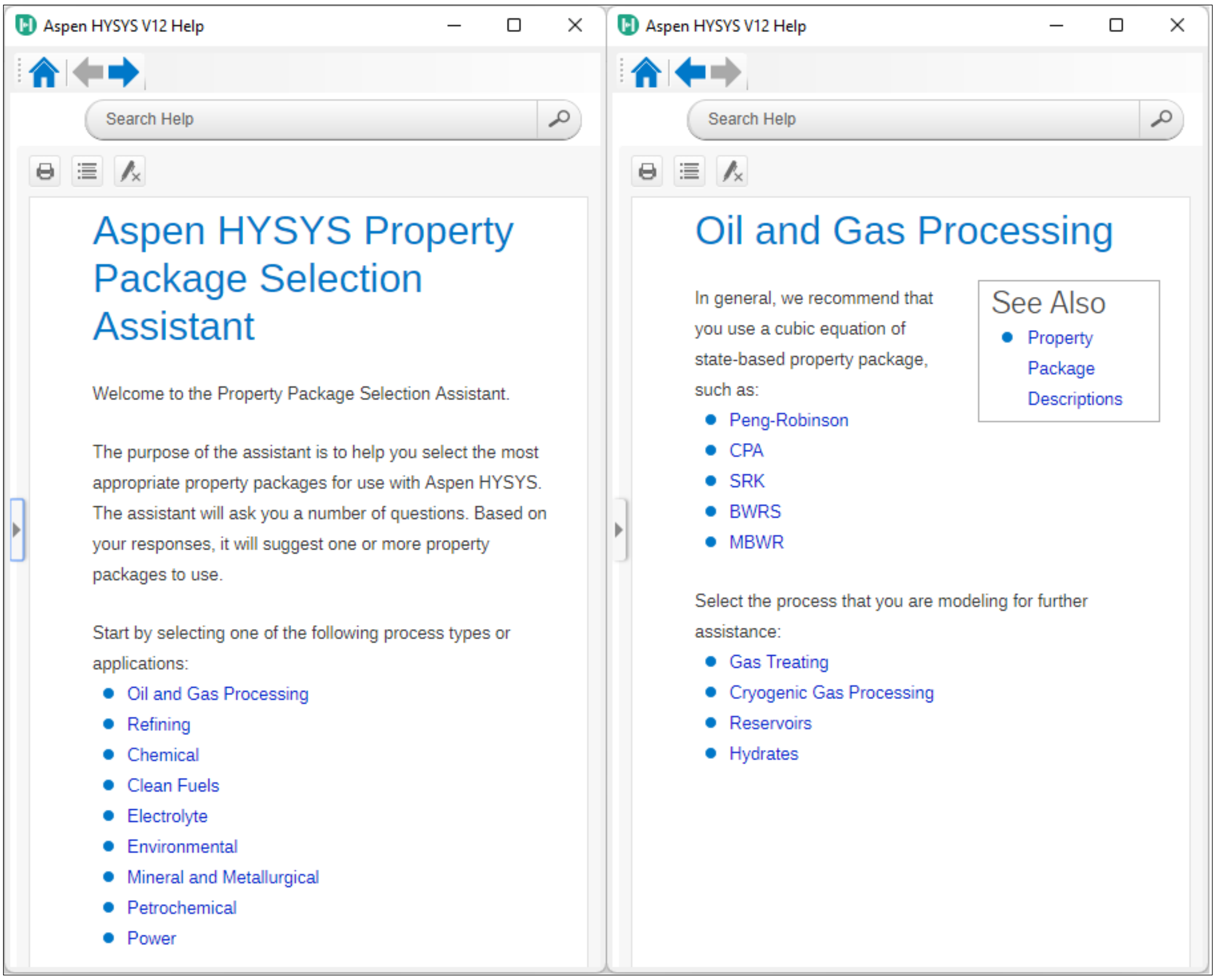
4.1.5. Model adjustment definition and assessment

The relative error was used to evaluate the models built along the modelling process. This is defined as relative to the design value (Equation (4.1).

$$RE[\%] = \frac{\hat{Y} - Y_D}{Y_D} \times 100 \qquad (4.1)$$

The **Model Adjustment Index (MAI)** is defined and proposed to assess and compare predictions made in all cases. This parameter quantifies the total deviation adding the individual error made predicting the value of each variable or operating condition.

Based in the **Mean Square Error (MSE)** and the **Root Mean Square Error (RMSE)** the MAI was defined as a ***Mean Square Relative Error (MSRE) (***(4.4).



Aspen HYSYS Property Package Selection Assistant

Welcome to the Property Package Selection Assistant.

The purpose of the assistant is to help you select the most appropriate property packages for use with Aspen HYSYS. The assistant will ask you a number of questions. Based on your responses, it will suggest one or more property packages to use.

Start by selecting one of the following process types or applications:

- Oil and Gas Processing
- Refining
- Chemical
- Clean Fuels
- Electrolyte
- Environmental
- Mineral and Metallurgical
- Petrochemical
- Power

Oil and Gas Processing

In general, we recommend that you use a cubic equation of state-based property package, such as:

- Peng-Robinson
- CPA
- SRK
- BWRS
- MBWR

See Also

- Property Package Descriptions

Select the process that you are modeling for further assistance:

- Gas Treating
- Cryogenic Gas Processing
- Reservoirs
- Hydrates

Figure 4.2. Aspen HYSYS Property Package Selection Assistant.

- Mean Square Error (MSE):

$$MSE = \frac{1}{n}\sum(\hat{Y} - Y)^2 \tag{4.2}$$

- Root Mean Square Error (RMSE):

$$RMSE = \sqrt{MSE} \tag{4.3}$$

- ***MAI=Mean Square Relative Error (MSRE)***:

$$MSRE = \frac{1}{n}\sum\left(\frac{\hat{Y} - Y_D}{Y_D}\right)^2 \tag{4.4}$$

- *Root Mean Square Error (RMSE):*

$$RMSRE = \sqrt{MSRE} \tag{4.5}$$

Variables compared include flows rate, compositions, temperatures, pressures, and other parameters. The magnitude in a deviation produced in a flow rate cannot be compared with a deviation in a composition. For this reason, the error was defined relative to the design value.

4.2. Real Plant Process Data

After selecting a reference product from the product portfolio different methods were boarded to establish the current operating conditions of the distillation section. The main source of data was the instrumentation records that are part of the Distributed Control System (DCS) providing historical values and others statistical data. The second source was the instrumentation that indicates and display values but do not have the feature of recording the measures. The last source was industrial and lab tests, and computer aided simulation.

All the procedures that involve analyse and process data from measured and controlled variables of the DCS (recorded measurements) were taken from the Aspen InfoPlus.21® (IP21) system using Aspen Process Explorer® to analyse and obtain statistical data.

Utilities consumptions were calculated based on a few assumptions. For the cooling water inlet and outlet conditions were easily obtained from the DCS records. For the low-pressure steam and refrigerant propylene, it was not possible to stablish the fraction of latent heat exchanged. Hence, the assumption of exchanging all the latent heat available was considered using this to calculate the total energy consumption for each utility.

Two version of the PFD are defined to compare and evaluate the performance with the original design. The first contains the design data and the second include the real plant data scenario. Those deviations from the original design were identified by comparing PDF leading to potential improvement suggestions.

Same analysed variables to compare models and cases of study during the modelling process were considered to assess the current operation of the distillation. To keep a data consistency, all the information from the several available sources was taken from the same period, to evaluate the same steady state.

4.2.1. Product portfolio evaluation

[redacted]. Some are specialties other commodities. Each product is made using a different recipe that generate a different carrier gas. Modifying the distillation feed conditions introduces variations in the column performance. Therefore, the best operating conditions for one product could not be the same for the others.

The product portfolio was evaluated to select one product. All test and trials to establish real plant data are performed during the manufacturing period of this reference product. Therefore, the improved case was suggested according to the data obtained for this product manufacturing conditions.

Reference product was selected by mutual agreement between the company tutor and other professionals from LYB TAO. This product belongs to the family of [redacted] and is manufactured several times in the year. This will provide representative conditions of the most frequent feeding conditions to the distillation section.

4.2.2. Experimental data

4.2.2.1. Composition Analysis

Main streams composition estimation was boarded by two approaches. The first estimate the composition considering the measured value of the mixture temperature. For bottoms and product streams was made considering a binary mixture to use the equilibrium diagram (propylene/propane system). The temperature of the mixture can provide an estimation of the composition. The chromatography instrumentation attached to the PPII distillation section is out service but the one installed in PPIII is working, the method first was applied in the PPIII C3-Splitter to assess the feasibility of the assumptions made.

The second approach was to send stream samples for further analysis in a lab of an external company. Five streams were selected (Figure 4.3) along with a defined list of components to analyse (Table 4.1).

[redacted]

Figure 4.3. Sampling points locations

After visiting the distillation section, the best locations for samples extraction were identified. Three of the five points need to be adapted to link the sampling nozzle and the container for sample storage. The connection point for the nozzle is available removing the installed manometers (P&ID tags PIXXXXX). The remaining two has already a sampling system installed (isolation valves and flanges).

In coordination with TQS, Production and Maintenance departments a specific date was selected to extract the samples, according to production schedule. These were collected by maintenance and quality staff following all the safety procedures. Finally, samples were tagged and sent to a third party to be analysed using the method ASTM D2163-14(2019). All the samples were taken from high pressure liquid streams and collected in special containers for high pressure liquids. Sampling period from the first sample extraction (12/20/2022 10:32:00) to the last (12/20/2023 12:52:00) lasted around 140 minutes.

Table 4.2. Sample extraction summary

[redacted]	[redacted]	[redacted]		[redacted]	[redacted]
		[redacted]	[redacted]		
[redacted]	[redacted]	[redacted]		[redacted]	[redacted]
[redacted]	[redacted]	[redacted]		[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

4.2.2.2. Vapour fraction

Estimation of the vapour fraction at the main condensers (W655A and B) outlet stream is made by industrial scale tests. Quantifying level variation produced in a specific period it is possible to estimate an average value of the liquid phase flow rate coming from the W655A/B condensers.

The test was designed in three stages to quantify the flow rate of the liquid and vapour phases coming from the top condensers. With few modifications the vent column was induced to operate as a phase separator and the flow meters attached to it were used to measure outlet streams flow rates.

The system considered is delimited by the control volume (CV) defined in Figure 4.4. This VC in composed by two types of fluids: a mixture of hydrocarbons and NCG, coming from the splitter (Carrier Gas) and hydrocarbons for refrigeration purposes (Refrigerant Propylene). Both fluids exchange energy with each other in condenser W659, without mixing.

[redacted]

Figure 4.4. Vapour fraction estimation test. Definitions.

Furthermore, two input streams (1 and 4) and three output streams (2, 3 and 5) pass through the control surface (CS). The steady state total mass balance is defined in Equation (4.6), where Gi is the total mass flow rate of stream i. Inlet and outlet flow of propylene refrigerant is the same, the total mass balance reduces to Equation (4.7). Flow meter [redacted] allow to read the flow rate of the reflux liquid stream, coming from the base of the reflux drum (G2). On the other hand, [redacted] provide the outlet stream flow rate leaving the [redacted] (G3).

$$G1 + G4 = G2 + G3 + G5, Gi\ \mathit{[kg/h]} \tag{4.6}$$

$$G4 = G5 \Rightarrow \boldsymbol{G1 = G2 + G3} \tag{4.7}$$

The internal streams in the [redacted] are analysed in Figure 4.5. In absence of refrigerant propylene flowing inside the condenser a new steady state is created where no liquid phase will return from the [redacted] (L1=0). Under these conditions, the reflux from the bottom of the reflux drum is the liquid phase (G1L) and the outlet stream at the top of the column will be the vapor phase (G1V) coming from the [redacted], respectively. Dividing the last by G1 the vapor fraction is obtained (Equation (4.13).

Another alternative based in the reflux drum level variation is boarded for vapour fraction estimation. Closing the reflux valve linked to [redacted] totally or partially, still in absence of refrigeration, a transient state with liquid phase accumulation occurs in the tank and is caused by G1L flow rate. Vapour stream has no influence and will find his way out through the vent column.

$$Vapour\ fraction\ (VF) = t_V = \frac{\boldsymbol{G_{1V}}}{\boldsymbol{G1}} = \frac{\boldsymbol{G3}}{\boldsymbol{G2 + G3}} \tag{4.8}$$

[redacted]

Figure 4.5. Liquid and vapour phases distribution inside the reflux drum (B655).

Total mass balance in the transient state is defined by the Equation (4.9). Without propylene refrigerant fed into condenser W659 the liquid phase coming from the bottom of the vent column is zero (L1=0). Furthermore, if the reflux is totally or partially restricted, the outlet flow rate has a lower value than the initial flow without reducing inlet stream flow rate. This cause liquid phase accumulation in the system until it reaches a new steady state. Mass balance is reduced then to the Equation (4.10). Considering a time-weighted average value of G1L, last expression can be simplified into Equation (4.11). The outlet stream from the reflux drum is only reduced by a fifty percent to prevent the trays from drying out.

$$\boldsymbol{G_{1L} + L_1 - G_2 = \frac{dM}{dt}} \tag{4.9}$$

$$\begin{gathered}\boldsymbol{\xRightarrow[\substack{L_1=0\\ G_2=G_{2\prime}}]{} G_{1L} - G_{2\prime} = \frac{dM}{dt} \Rightarrow \int_{M0}^{M1} dM = \overline{G_{1L} - G_2{}'} \int_{t_0}^{t_1} dt}\\ \boldsymbol{\Rightarrow M_1 - M_0 = \overline{G_{1L} - G_2{}'}(t_1 - t_0)}\end{gathered} \tag{4.10}$$

$$\boldsymbol{\Rightarrow G_{1L} = \rho \frac{VT_1|_{h_1} - VT_0|_{h_0}}{t_1 - t_0} + G_2{}'} \tag{4.11}$$

As said previously, the data collection was made in 3 stages setting previously the reflux drum level in 30%:

Stage 1. Initial steady state flow rates is made (process usual operating conditions). Measured value by FIRC 65105 and FIRA65107 flow meters are G2 and G3. Equation (4.7) provide the total flowrate leaving the condensers W655A/B (G1).

Stage 2. Propylene refrigerant supply to the W659 condenser is shutted off. Once the system stabilizes in a new steady state, the values read on the FIRC 65105 and FIRA65107 meters were G1L and G1V.

Stage 3. Close reflux feed valve to plate 100, FIRC65105. Simultaneously, the time elapsed is counted starting from the instant of valve complete closure to the moment of a maximum level of 70% of the maximum capacity of the B655 tank. Considering initial al final level, the mass of liquid accumulated in the test period is calculated. Using

Equation (4.11) provided the value of G1L to compare it with the previously obtained from the FIRC65105 meter in stage 2.

4.2.3. Data from DCS records

Aided by Aspen® Process Explorer and Microsoft Excel all the DCS data is compiled. The interval of time considered for statistical purposes is the period defined in Section 4.2.2. Average value of each variable was obtained and considered as the representative value of this period.

4.2.4. Data from in site instrumentation

Instant value of those variables that were measured and displayed on site but no recorded were registered as a photo for further processing. These photos were taken in the same period defined in section 4.2.3 during samples collection.

4.2.5. Data estimation aided by simulation

Gas phase feed composition was estimated using HYSYS®. The feed stream to the distillation columns [redacted]) (Figure 4.6). Both streams have individual flow meters and are sent directly to [redacted]). The composition of the liquid feed stream was obtained by lab tests not being possible to replicate this methodology for the vapour phase.

This phase separation occurs in a [redacted]) that condenses the feed stream to the column and is placed upstream the distillation section. This separation was simulated assuming both phases are in equilibrium. The objective was to estimate the composition of the vapour phase in equilibrium with a liquid phase with the composition obtained in the laboratory analysis. Once the composition was estimated, considering the density of the gas phase and measured value reported by the flow meter installed, the feed stream total mass flow was calculated.

[redacted]

Figure 4.6. Distillation feed stream (carrier gas)

4.3. Comparison Design with Current Operating Conditions

Once all the needed data is compiled, the current version of the PFD is finally defined. Then, a final comparison of both steady states conditions (design and real plant) was easily performed. The deviations from the design were highlighted and assessed to finally include it in this report conclusions and being discussed with the Review Meetings Team.

4.4. Model calibration with real plant data

The feed stream obtained from the real planta data is loaded in the model to compare the predicted outlets against the actual obtained in the process.

Comparison is made following the same methodology applied before to compare model predictions for the basic design engineering information. Predictions are calibrated varying the column global efficiency.

4.5. Improvement & Optimization

The base case built was improved with the help of HYSYS. A screening of key variables related with the column's performance was made and those variables that could be modified (the number of stages cannot be modified or changed) were assessed based on a cost-benefit basis. Then, the improved operating conditions were suggested to operate the columns.

4.5.1. Basics

System behaviour was analysed creating different cases of study in HYSYS. Then, for every key variable a feasible operation interval with the respective energy consumption was obtained. Furthermore, the cost of utilities was defined and calculated for the separation process along this interval.

Focusing on a costs and benefits analysis, an improved interval was obtained to operate the columns. In case of controlled variables this provide better set points for controllers.

4.5.2. Reflux rate effect

The operation of distillation columns was linked naturally to some key variables. Among them and due to time limitations, the main key variable evaluated is the reflux ratio.

The effect of the reflux ratio (or reflux rate) was analysed boarding a theoretical and an empirical approach. Using the model, the system behaviour was predicted defining a case of study in HYSYS®. It was desired to analyse the effect on key process variables:

[redacted]

For the empirical analysis, the methodology intended to be similar but must be modified. The chromatography analysers attached to the distillation section were out of service not being possible to stablish the distilled product composition directly. For this reason, the reaction section analysers were used to assess distillate composition. Despite some limitations related to the age of the instrumentation, these were calibrated and in service during the execution of this project. Then, the new methodology integrates both sections allowing also to analyse the effect of the distillation performance over the reaction performance.

Using a black box approach, the raw material feed into the reactor is a mix of [redacted] (provided by [redacted]) and [redacted] (Figure 4.7). The composition of the reaction atmosphere is the results of these two feed streams. Therefore, the amount of impurities inside the reactor are mainly related to the distillation performance and the distillate composition.

Reaction rate for gas phases reactions is directly affected, among others, by reactants partial pressure. Assuming the same reaction conditions, for higher amount of propane introduced in the reactor lower reaction rate will be experienced due to lower propylene partial pressure. Therefore, is preferably to introduce the lower amount of propane in the reactor.

[redacted]

Figure 4.7. Reaction and distillation sections feed streams.

The analysers calibration was checked in a preliminary phase. The findings were that the error measuring propylene composition was higher than for propane (Table 4.3). Both compositions are related to the distillation performance and either can be used to conclude about the distillation performance. The best separation performance is obtained for a distilled stream more concentrated in propylene with traces of propane. Increasing propane composition in the distillate decrease propylene recovered and the performance decrease. Finally, propane composition in the reaction atmosphere was analysed instead propylene composition.

Empirical tests were performed during the columns operation in normal conditions and was divided in two phases. The 1st Phase test were performed in a smaller interval of reflux ratio. The objective was to evaluate the performance of the reboiler purge system response for small increases in the reflux rate and confirm theoretical statements. The test started on January 16th at 22:00:00 and finished on January 19th at 21:55:00. Three products from the family of [redacted] are scheduled to manufacture in this period: [redacted]. Reflux ratio values analysed went from [redacted] (normal operating condition) to [redacted]. Variables analysed are defined in Table 4.4.

Table 4.3. Preventive maintenance and calibration sheet. Analysers [redacted].

[redacted]	[redacted]		[redacted]	
	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]		

The 2nd phase test includes higher values of reflux ratio during a longer period. The test started on February 6th at 8:00:00 and finished on February 10th at 1:20:00. Two products from the family of [redacted] are scheduled to manufacture in this period: [redacted]. Reflux ratio values went from [redacted]. Variables analysed are defined in Table 4.5.

Table 4.4. 1st Phase test variables analysed.

	Variable P&ID tag	**Description**
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]

4.5.3. Optimization

Optimal reflux rate is obtained with the HYSYS® Optimizer module (appendix A3). The minimum required data was the objective function and constrains and boundaries for every primary variable. The objective function is introduced in Equation (5.1 and the primary variables selected in Table 4.6.

$$Benefit = Incomes - Outcomes$$
$$= P_{PROPYLENE} \times (w_{PROPYLENE} \times G)_{PRODUCT} \times t_{OP} - \sum_{i=1}^{3} k_i Q_i \quad (4.12)$$

Table 4.5. 2nd Phase test variables analysed

	Variable P&ID Tag	Description
1	[redacted]	[redacted]
2	[redacted]	[redacted]
3	[redacted]	[redacted]
4	[redacted]	[redacted]
5	[redacted]	[redacted]
6	[redacted]	[redacted]
7	[redacted]	[redacted]
8	[redacted]	[redacted]
9	[redacted]	[redacted]
10	[redacted]	[redacted]
11	[redacted]	[redacted]
12	[redacted]	[redacted]
13	[redacted]	[redacted]
14	[redacted]	[redacted]
15	[redacted]	[redacted]
16	[redacted]	[redacted]
17	[redacted]	[redacted]
18	[redacted]	[redacted]

Table 4.6. Primary variables summary.

Variable	Description	Upper bound	Lower bound
$x_{PROPYLENE,PRODUCT}$	Propylene purity in product distilled stream	[redacted]	[redacted]
$x_{PROPANE,BOTTOMS}$	Propane purity in bottoms stream	[redacted]	[redacted]

The product and bottom streams purities boundaries comes from the basic engineering design. The reflux rate has lower and upper bound when defining the feasible region and which values analyse when the optimum screening is performed. The lower bound is taken from the shortcut distillation using the minimum reflux as a reference to estimate the minimum flow rate. The upper bound is provided by the maximum flow rate defined in the columns datasheets which should correspond to the 80% of the flooding flow rate.

The propylene price is extracted from the company annual reports considering the [redacted] directly from the provider [redacted]. The cost of the utility i associated to the duty i was estimated with a general expression composed by coefficients A, B, and C defined in Equation (4.13. The last, can be reduced into a simpler expression introduced by Equation (4.14 where all constants are grouped into one constant ki. Units and values of each coefficient are defined in

Table 4.7. Additional and detailed information about utilities cost and assumptions made can be found in Appendix A5.

$$C_i = A_iB_iC_iQ_it_{OP}, i = 1, 2, 3 \ \cap \ t_{OP} = \text{[redacted]}\ {}^{h}/_{año} \tag{4.13}$$

$$C_i = k_iQ_i, [k_i] = {}^{€}/_{Año \times kW}, [Q_i] = kW \tag{4.14}$$

Table 4.7. Utilities summary

Utility	Coefficients			
	A	B	C	k
[redacted]	[redacted] [redacted]	[redacted] [redacted]	[redacted] [redacted]	[redacted]
[redacted]	[redacted] [redacted]	[redacted] [redacted]	[redacted] [redacted]	[redacted]
[redacted]	[redacted] [redacted]	[redacted] [redacted]	[redacted] [redacted]	[redacted]

5. RESULTS AND DISCUSSION

5.1. Process Modelling & Simulation

5.1.1. Basic Engineering Design

[redacted] (5.1).

Table 5.1. C3 Splitter design conditions

[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]

(a): [redacted]

(b): Detail extracted from Figure 5.1

$Reflux\ ratio^{(a)} =$ [redacted] (5.1)

[redacted]

Figure 5.1. Reboiler configuration. Drawings constructive specifications.

[redacted]

Figure 5.2. Constructive details of tray [redacted] [redacted].

The design feed components are described in Table 5.1. Based on the feed flow rate, composition, and properties and the components defining the system (Table 5.3) the shortcut distillation module estimates minimum reflux ratio (Underwood), minimum number of stages (Winn) and actual number of stages (theoretical number of plates with Gilliland), using Winn, Underwood, and Gilliland correlations. Light key (LK) and heavy key (HK) components are [redacted]. The input loaded is shown in

Table 5.4 with the LK and HK recoveries in distillate stream obtained from the PFD and the reflux ratio from Table 5.1. Results are presented in Table 5.5. Using these results and Equation (1.1 the design [redacted]%.

Table 5.2. Design feed components.

[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

Table 5.3. Design feed flow rates, composition, and properties.

Component	**Flow rate [kg/h]**	**Mass fraction**
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]

[redacted]	[redacted]
[redacted]	[redacted]

The splitter [redacted]) is designed to remove light hydrocarbons, nitrogen, hydrogen, carbon monoxide and carbon dioxide. On the other hand, is provided with a [redacted]) [redacted] using [redacted]. The design specifications are introduced in Table 5.6.

The utilities involved in the distillation operation are three:

[redacted]

Table 5.4. Shortcut distillation input. Design data.

Input	Value
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]

Table 5.5. Shortcut distillation results.

[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]

Table 5.7 show the temperature profile extracted from the basic engineering design of the [redacted]. Table 5.8 summarize all the basic

engineering the data compilation. The design case is then fully defined introducing streams conditions and compositions, utilities consumption and the expected distillation performance.

Table 5.6. Vent column design conditions

[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]

Table 5.7. Distillation column temperature profile. Design data.

[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]

Temperature [ºC]

[redacted]

Tray Nº

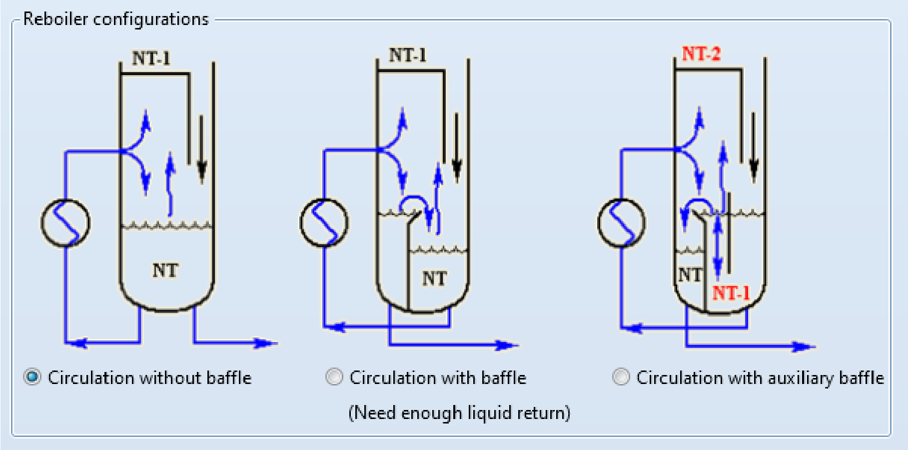
Figure 5.3. Distillation column temperature profile. Design data.

Table 5.8. Process Flow Diagram Data. Design version.

[redacted]

5.1.2. Modelling

Distillation module following the HYSYS® usual procedure were easily followed based on the information compiled in the previous section. The real reboiler configuration could not be adopted in the model. Despite the strategies tried it was not possible to converge the flowsheet selecting the [redacted] option. Therefore, the default option was selected: *circulation without baffle* (Figure 5.4). Flowsheet obtained from the first step model is introduced in Figure 5.5.



● Circulation without baffle
○ Circulation with baffle (Need enough liquid return)
○ Circulation with auxiliary baffle

Figure 5.4 – HYSYS options for reboiler configuration.

To build the second step model flowsheet other convergence issues appeared. The addition of the vent column to the first model implies to include, among other modifications, the liquid stream from the bottoms of the column to the reflux drum. This stream is a recycle stream with a recycle module linked to it. Connecting this recycle module and to converge the flowsheet with this configuration was the main challenge.

[redacted]

Figure 5.5. First step model flowsheet and subflowsheet.

Several alternatives were tested to converge the flowsheet:

- ✓ Define the vent column inside the column build environment. In that sub-flowsheet space, there is no recycle module. Instead, the algorithm must be changed from HYSIM Inside-Out to Modified HYSIM Inside-Out.
- ✓ Keep the vent column inside the column build environment but sending the streams to a recycle module outside in the main simulation environment.
- ✓ Define the vent column and the recycle in the main simulation keeping the default condenser in the main tower module.

The problem was identified at the top of the column when trying to estimate the top vapor stream properties, especially the outlet temperature. Properties estimations are related to the fluid package selected (FP). Therefore, other options of fluid packages were added to test and analyse the influence of these when trying to predict the properties of the top vapor stream. Alternatives of FP analysed were taken from the HYSYS help mainly and others suggested in the literature studying the behaviour of propane/propylene systems. The top vapour stream composition was also tested comparing two types of streams including or not the NCG (Table 5.9).

Table 5.9. Top vapour composition. FP Test Nº1.

	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]
[redacted]		[redacted]	
[redacted]	[redacted]	[redacted]	

Using the design data, the Test Nº1 stablished and analysed:

- Top vapour stream vapour fraction (VF) predicted by the model.
- Condenser outlet stream vapour fraction and duty. This is made imposing that the top vapour temperature drop through the condenser is the same that the one in the design [redacted]).

Flowsheet created for fluid package test Nº1 is introduced in Figure 5.6 and the results obtained can be seen in Table 5.10. The top vapour fraction predicted varies according to the fluid package considered. The Cubic-Plus-Association (CPA) fluid package estimated the proper fraction that the top vapour stream should have after leaving a distillation column. Including the NCG increase the vapour fraction due to the addition of these components mass flow rates are directly to the vapour phase. If NCG are not included the vapour fraction at the condenser outlet is zero despite the FP analysed. Including the NCG some FP predict a vapour fraction higher that zero at the condenser outlet. This is due to the presence of hydrogen and nitrogen that cannot be condensed with cooling water at these conditions. The amount of vapor predicted at the inlet varies the condenser duty. Higher vapour fractions predicted require higher duty to produce the temperature drop (-4,2ºC).

Test Nº2 evaluate the predictions made by a selected group of fluid packages: PR, SRK and BWRS. CPA is not feasible for the system under study and is not further included. All the components defined in the design top vapour stream were included (Table 5.1). The top vapour stream leaving the distillation column has no C4 or heavy HC, these compounds cannot be found in this stream. The vapour fraction predicted is also analysed but with other considerations and methodology.

[redacted]

Figure 5.6. Fluid package and feed stream composition test. HYSYS® flowsheet. FP Test Nº1.

The temperatures defined by design are imposed:

- TA=Top vapour temperature. Source: LYB ‘s Process Flow Diagram.
- TB=Reflux temperature. Source: LYB ‘s Process Flow Diagram.
- TC=Top vapour temperature. Source [redacted] datasheet
- TD=[redacted] outlet stream temperature. Source [redacted] datasheet

The analysis is based on evaluate the predictions made by the software varying stream composition and the FP used to predict the properties.

The alternatives of streams analysed are shown in Table 5.11 and the results inTable 5.12. In the last, dew and bubble point temperature were estimated and the difference between both calculated. Then, for all the temperatures defined by design the vapour fraction is predicted.

Table 5.10. Predicted top vapour fraction and utility consumption. FP Test Nº1.

[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

Table 5.11. Streams composition alternatives. FP Test Nº2.

		[redacted]	
[redacted]		[redacted]	
	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]

Each FP predict different values for every stream and vary considering the components included. From stream 1 to stream 3, lighter components are added causing:

- ✓ Smaller dew and bubble points because lower boiling points are included in the mixture temperature calculation.
- ✓ Higher temperature difference between these two temperatures.
- ✓ Higher vapour fractions predicted.
- ✓ Analysing the FP effect, PR and BWRS in general predict similar values while SRK values are far from those.

Considering FP and components included, the most accurate prediction comes from PR and assuming the top vapour stream composed by all the components previously mentioned. The design vapour fraction is obtained from the condenser data sheets (Figure 5.7 and Equation (5.2).

Table 5.12. Dew and bubble point temperatures and vapour fraction predictions. FP Test Nº2.

			[redacted]			[redacted]			
			[redacted]			[redacted]			
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
			[redacted]	[redacted]		[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

[redacted]

Figure 5.7. Design vapour fraction. [redacted] data sheet.

[redacted] (5.2)

Going back to the model building procedure, after trying different alternatives, the flowsheet defined for the second step model converge and the initial base case is sucesfully built (Figure 5.8).

[redacted]

Figure 5.8. Initial base case (second step model).

5.1.3. Models’ predictions

Model predictions are defined using a black box approach. Inlet and outlet streams such as feed, vent gas, product and bottoms are considered external streams and top vapour, and reflux are internal streams.

For the first model, the feed stream loaded is defined in Table 5.13. Internal streams flow rate and mass fractions results are introduced in Table 5.14 and Table 5.15. The first model is capable to predict the internal streams flow rate with high accuracy. Both predictions have a very low relative error. In the case of mass fractions, the model is not so accurate predicting internal stream with lower concentrations of propylene and higher concentrations of ethylene. Propane content is not so different from the design.

External streams flow rate and mass fractions are included in Table 5.16 and Table 5.17. Flow rate predictions have very low relative error but mass fractions present considerable deviations specially for the propane content in product stream and propylene in bottoms. The other mass fractions predicted have relative error that goes from 5 to 10%.

The predictions of other design specifications and key variables presented in Table 5.18 are very similar to the ones defined in the basic engineering design except the duty requirements. The energy estimated both in the condensers and in the reboiler are lower for the model that the defined in the basic engineering design.

These steady estate conditions are the starting point for the definitive model that includes the vent column attached to the [redacted].

Table 5.13. First model feed stream.

	[redacted]	
[redacted]		
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	
[redacted]	[redacted]	
[redacted]	[redacted]	
[redacted]	[redacted]	

Table 5.14. First model internal streams flow rate predictions.

[redacted]	[redacted]		[redacted]
	[redacted]	[redacted]	
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]

Table 5.15. First model internal streams mass fractions predictions.

[redacted]	[redacted]	[redacted]		[redacted]
		[redacted]	[redacted]	
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]

Table 5.16. First model external streams flow rate predictions.

[redacted]	[redacted]		[redacted]
	[redacted]	[redacted]	
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]

Table 5.17. First model external streams mass fractions predictions.

[redacted]	[redacted]	[redacted]		[redacted]
		[redacted]	[redacted]	
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]

Table 5.18. First model design specifications and other key variables.

[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]

Next, the base case is tested. For this, three types of feed streams are defined and loaded in the model to check the predictions obtained. Each type defines one case of study (Table 5.19) and is basically characterized by the type of components included:

✓ Feed Stream A (FSA)=Main hydrocarbons
✓ FSB=Main hydrocarbons+Non-Condensable Gases (NCG)
✓ FSC=Main hydrocarbons+NCG+C4-Hydrocarbons+[redacted]

Table 5.19. Feed streams flow rates and composition for the base case model.

	[redacted]		[redacted]		[redacted]	
[redacted]	[redacted] [redacted]	[redacted] [redacted]	[redacted] [redacted]	[redacted] [redacted]	[redacted] [redacted]	[redacted] [redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]			[redacted]	[redacted]	[redacted]	[redacted]
[redacted]			[redacted]	[redacted]	[redacted]	[redacted]
[redacted]					[redacted]	[redacted]
[redacted]					[redacted]	[redacted]
[redacted]					[redacted]	[redacted]
[redacted]	[redacted]		[redacted]		[redacted]	

The case of study A easily get to a steady state solution. The internal streams flow rate results (Table 5.20) are very similar to the design flows and without meaningful variations between all the FP analysed. Each prediction relative error (relative to the design value) is reported in parenthesis to the right of the model estimation. Internal streams mass fractions (Table 5.21) are also quite similar for both streams and almost all the FP. The best fit is obtained with the PR FP and the higher deviation is produced using the BWRS FP.

Product and bottom predicted flow rates are similar to the design. Vent gas flow rate is different introducing a higher deviation from the design (Table 5.23). For all the FP analysed the base case model predict almost the same flow rate for the last. The results obtained for mass fractions vary for each stream and FP. The product composition (Table 5.22) is very accurate with the best fit produced by SRK FP followed by PRSV, PR and the finally BWRS that has the highest relative error. Bottom stream composition is well predicted only by SRK FP, the others are far from the design. Vent gas composition has the highest relative error for compositions predictions among all external streams.

The specifications and key variables predicted are mostly different from the design (Table 5.24). Only the reboiler duty, the top vapour temperature, the reflux ratio, the propylene mass recovery are predicted with low relative error (less than 5%). The other variables and parameters are predicted with a high relative error (more than 15%).

For the case of study with the feed stream option B, it was not possible to converge the flowsheet using the BWRS FP. Therefore, no results are reported for this alternative. The predictions obtained for the internal streams flow rate and composition are very good (Table 5.25 and Table 5.26). The flow rates are predicted identically by all the FP analysed. The compositions results show very good results for all FP in general with the best predictions obtained with PR.

The external streams flow rates and compositions for case B have less relative error compared to the option A (Table 5.27 and Table 5.28). For all flow rates the three FP alternatives predict results around the same value. For the mass fractions, some values are best fitted by PR and other by SRK.

Specifications and design variables are estimated with less error as can be seen in Table 5.29. The reboiler duty, the top vapour temperature, the reflux ratio, and the propylene mass recovery has the best fit of all the analysed variables with an error less than 5%.

For the last case of study including feed streams option C, the internal streams flow rates are very good predicted by the model showing a relative error less than 0,5% (Table 5.32). Mass fractions are not the best predictions of all cases and the relative error reported is the highest, especially using BWRS as FP.

External streams flow rates (Table 5.32) in this case of study are also very well predicted. Product mass fractions predictions have an acceptable fit except for the added components in this case of study (methane and ethane). SRK predict the more accurate values and BWRS report the higher deviation. For the vent gas stream, the estimations are very good, except for predicting the propane content. For this stream PR seems to have the best results and BWRS report higher deviation. For the bottom stream SRK have a very good fitting and the others FP are not so accurate.

Results obtained estimating the other design specs and variables report that only for the reboiler duty, the top vapour temperature in K655, the bottom temperature, reflux ratio, propylene mass recovery, and propylene mass flow rate in vent gas the model predict values with a relative error less than 5%. The other variables and parameters have higher deviation from the design.

Table 5.20. Internal streams flow rate predictions. Feed stream A.

[redacted]

Table 5.22. External streams predicted composition. Feed stream A.

[redacted]

Table 5.23. External streams flow rate predictions. Feed stream A.

[redacted]

Table 5.24. Design specifications and others key variables. Column K655A/B and vent column K656. Feed stream A.

[redacted]

Table 5.25. Internal streams flow rate predictions. Feed stream B.

[redacted]

Table 5.26. Internal streams predicted composition. Feed stream B.

[redacted]

Table 5.28. External streams flow rate predictions. Feed stream B.

[redacted]

Table 5.27. External streams predicted composition. Feed stream B.

[redacted]

Table 5.29. Design specifications and others key variables. Column K655A/B and vent column K656. Feed stream B.

[redacted]

Table 5.30. Internal streams flow rate predictions. Feed stream C.

[redacted]

Table 5.31. Internal streams predicted composition. Feed stream C.

[redacted]

Table 5.32. External streams flow rate predictions. Feed stream C.

[redacted]

Table 5.33. External streams predicted composition. Feed stream C.

[redacted]

Table 5.34. Design specifications and others key variables. Column K655A/B and vent column K656. Feed stream C.

[redacted]

5.1.4. Model adjustment

Table 5.35 and Table 5.36 report the error obtained modelling the three cases of study. The first is a comparison between Mean Square Error that is usually used to assess model's fitting and an alternative Mean Square Relative Error. For the MSE, the magnitude order difference can be noted compared to the MSRE (MAI) that reduce this difference when comparing a deviation from the design in a flow rate and a deviation in a bounded variable as a mass fraction. These results show that the MAI reduce these differences and provide a better evaluation of the error introduced by different types of variables. Analysing the MAI, the option with the lower value is the B, and the FP that introduces the minimum error is PR.

Comparing results obtained from the Root Mean Square Error (RMSE) and Root Mean Square Relative Error (RMSRE) the conclusions are similar: the lower value is obtained for the option B using PR as the FP. Here, can be noted the same effect previously mentioned using absolute error or relative error to compute the model adjustment comparing different types of variables (bounded and unbounded ones).

Table 5.35. Model adjustment. Comparison MSE and MSRE.

[redacted]	[redacted]				[redacted]			
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

Table 5.36. Model adjustment. Comparison RMSE and RMSRE.

[redacted]	[redacted]				[redacted]			
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

With the best model base case, the design temperature profile per stage is built and can be seen in Figure 5.9. PR, SRK and PRSV predicts a profile very similar which for trays located upper the 50 matches with the design profile but for lower trays is not the case. BWRS profile trend for the first [redacted] trays is similar to the design but does not match in values.

[redacted]

Figure 5.9. Case of Study B. Column temperature profile.

5.2. Real Plant Data

5.2.1. Experimental results

The results obtained for composition estimation based on the mixture temperature are shown in Table 5.37. The PPIII C3-Splitter operating pressure is about 10 barg and the true boiling point temperature (TBP) for pure components at this pressure is included at the top of the table. Right below these data, the mixture temperature reported by the DCS for bottom and product streams, the composition measures reported by the gas chromatography (GC) analysers, and the values estimated using the binary mixture equilibrium diagram are reported. Figure 5.10 show the methodology applied for the mixture temperature measured in the product stream. The temperature is [redacted]ºC and with the help of equilibrium diagram from Aspen Plus® the composition of the mixture is graphically obtained in mole fraction (Figure 5.10). The relative error is reported in parenthesis below the estimated value. In general, the method is not very accurate predicting the compositions. Compared with the concentrations reported by the GC analysers the error reported by this method is high. This approach presents the disadvantage that in the bottoms, other components are present being not possible to assume a binary mixture. Applying this method in PPII, the mixture temperature was located out of the interval defined between the pure components boiling point.

The second approach results are introduced in Table 5.37. The methodology report compositions in mole fractions for every sample provided and previously codified. It is important to note that the splitter liquid feed stream ([redacted]) has a mole fraction of [redacted]%m/m. The [redacted] stages in the main tower at the real operation condition produces a distilled with a [redacted]%m/m meaning that the splitter increase the propylene fraction only by [redacted]%. In a distillation column in which the product is extracted by the top and comes from the same reflux drum, the main product content is the same in the distilled stream and in the reflux stream. In this case, the distilled product is a side stream drawn off from tray [redacted] after introducing the reflux in tray [redacted]. Observe that the reflux (R100-01) mole fraction is [redacted]% and in the product is [redacted]%. This means that in 5 trays, that liquid stream is capable to increase the propylene content from [redacted]% to [redacted]%.

Table 5.37. Composition estimation from mixture temperature in PPIII.

[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]

		[redacted]	[redacted]		[redacted]	
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
			[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
			[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
			[redacted]	[redacted]	[redacted]	[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
			[redacted]	[redacted]	[redacted]	[redacted]

[redacted]

Figure 5.10. Composition estimated based on the mixture temperature in product stream. Mole fraction.

Results also show that the main tower bottom contain water, isopropanol and mineral oil. These three components increase this stage bubble point temperature making not possible to assume a binary mixture to estimate the composition of bottom stream. Almost in all streams, propane and propylene fractions summation gives more than [redacted]. The exception is observed in the reflux stream from main tower condensers to tray [redacted] and bottom stream. Results showing the note ‘Out of sample’ means that the sample was volume was not enough for the determination of the component analysed.

Table 5.38. Composition analysis results

		[redacted]				
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

[redacted]

[redacted]

5.2.2. DCS data

The main source of information to characterize real process was the recorded DCS historical data. Aspen Process Explorer® provided all the historic values to process it and calculate the required statistical information.

Figure 5.11 and Figure 5.12 show results obtained processing data using both softwares. The first plots the recorded measured values including the average and standard deviation associated to the steady stated during the sampling period. The second plots the same variables trend during the sampling period but the statistical data is reported below in a summary table showing average and standard deviation among others. As can be seen, the vapour feed stream flow rate was in average of [redacted] [m^3/h] with a standard deviation of [redacted] and the liquid flow at [redacted] [m^3/h] with a standard deviation of [redacted].

Flow rate [m3/h]

[redacted]

Figure 5.11. Feed streams flow rate to distillation column. Microsoft Excel® results.

[redacted]

Figure 5.12. Feed streams flow rate to distillation column. Aspen Process Explorer® results.

The distillation column temperature profile was obtained from the DCS records. The average values obtained during the sampling period reported in Table 5.39 and Figure 5.13.

Table 5.39. Distillation column temperature profile. Real plant data.

[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]

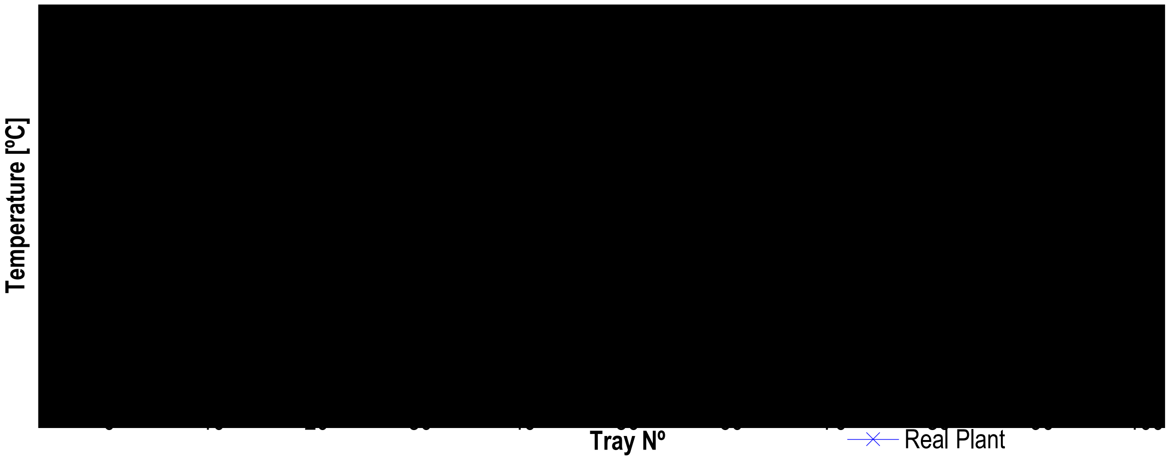

[redacted]

Figure 5.13. Distillation column temperature profile. Real plant data.

The same methodology was boarded to extract from the InfoPlus.21® servers all the other variables needed (Table 5.8) to build the PFD real plant version.

5.2.3. Utilities estimation

Estimate utilities during the sampling period was possible. Utilities exchanging sensible heat (cooling water) was the easiest to estimate but those exchanging latent heat (low pressure steam and refrigeration propylene) was made under a main assumption.

The duty related to the cooling water was a simple and direct calculation using inlet and outlet temperatures and flow rate. These three variables' values were extracted from the DCS records. For the other two, it was important to stablish if all the available latent heat was exchanged or not analysing the outlet stream to compute it in the calculations. For the low-pressure stream, the determination of the outlet temperature as an indication of fully condensed or subcooled liquid at the outlet of the reboiler was boarded. The outlet stream has no temperature measurement but for turbulent flow inside metal pipes, wall temperature is very close to the fluid outlet temperature. Using the instrument showed in Figure 5.14 the wall temperature was measured obtaining at the inlet around [redacted]ºC and at the outlet around [redacted]ºC. Low pressure steam ([redacted]) has a temperature of [redacted]ºC, and the temperature measured cannot be used to estimate the vapour title at the outlet. Then, after this preliminary test, both utilities were calculated assuming that all the available laten heat was exchanged during the operation.

Table 5.40, Table 5.41 and Table 5.42 report the results obtained calculating the real plant duty. The main tower condenser is composed by two heat exchangers, A and B, each with thermocouples installed at the inlet and outlet cooling waters streams. Both exchange a total heat of [redacted]kW. The reboiler duty during the sample extraction was around [redacted]kW and the vent condenser duty of [redacted]kW.

All the data used to estimate the latent heat of every fluid at the operating conditions were extracted from NIST database.

[redacted]

Figure 5.14. Wall temperature measurement. (a) Surface probe. (b) W656 reboiler.

Table 5.40. Cooling water consumption. Main condenser real plant duty.

	[redacted]		
[redacted]	[redacted]		[redacted]
[redacted]		[redacted]	
[redacted]		[redacted]	
[redacted]	[redacted]		[redacted]
[redacted]		[redacted]	
[redacted]		[redacted]	
[redacted]	[redacted]		[redacted]
[redacted]		[redacted]	

Table 5.41. Low pressure steam consumption. Reboiler real plant duty.

[redacted]	
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]

Table 5.42. Propylene refrigerant consumption. Vent condenser real plant duty.

[redacted]	
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]

5.2.4. Condensers outlet vapour fraction estimation

Vapour fraction at the condenser outlet is a relevant parameter to check. In the modelling process it was noticed that this value varies depending on the FP selected to predict the properties in this particular system composed by two components to separate with a very close relative volatility.

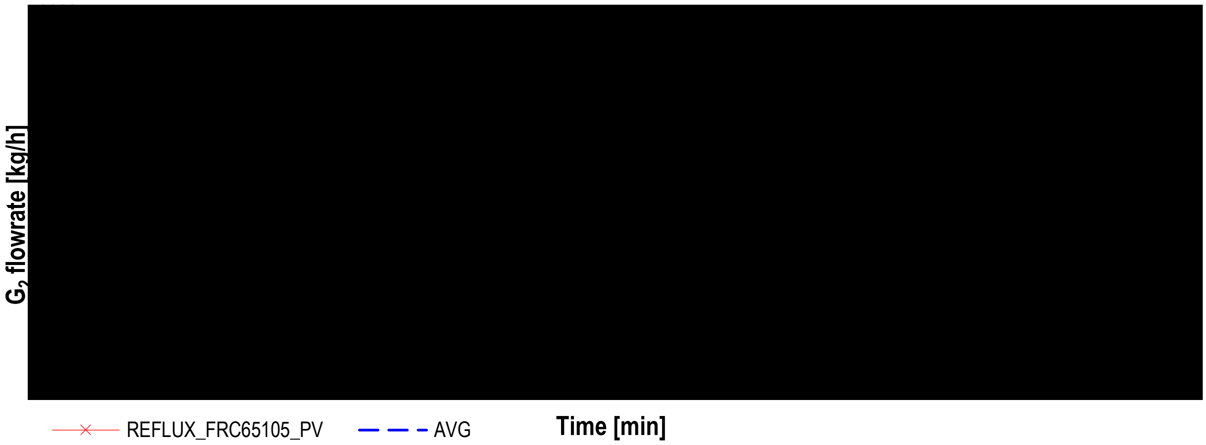
Stage 1 and 2 results can be seen in Figure 5.15 and Figure 5.16. Here, the time evolution of the G2 and G3 are introduced. Markers in red shown the data measured and recorded by the flowmeters. The dashed blue line represents the average value for each variable during the steady state period. Analysing the plots, the reflux flowrate

(G2) remains practically stable during the test. On the other hand, vent gas stream goes under a transient state starting in zero (stage 1) to finally reach an approximately constant value (new steady state in stage 2). G2 in stage 2 and 3 has the same value: [redacted] [kg/h]. G3 in stage 1 in the period analysed is zero and in stage 2 is [redacted] [kg/h].

The vapour fraction obtained is included in Figure 5.16. Comparing this with the design, the condensers in real plant exchange a higher amount of heat condensing more vapour.

The stage 3 is more detailed and involves more variables than stage 2 and is characterized by a bigger group of variables. Figure 5.17 define variables involved and instrumentation installed. Figure 5.18 show the time evolution of all the variables in one plot using Microsoft Excel® and in Figure 5.19 the same data but from Aspen Process Explorer®.

In Figure 5.18 the whole process time evolution can be identified. First, the initial conditions were the reflux drum level at the [redacted]. Previous night, the reflux level was decreased from the usual value of [redacted]% to have a higher range to increase it during the stage 3 (from [redacted]%). Then, between 6:15 and 6:30 the propylene refrigerant valve is fully closed (the instrumentation reports a signal higher than zero but the valve was completely closed). Around 8:00, the system reaches thermal equilibrium reporting both outlet streams temperature the same value (Figure 5.20). Without refrigerant flowing inside the vent column condenser, the vapour stream is no longer going back as a liquid stream to the column and leaves the column by the top. Around 9:00 the vent gas stream flowmeter starts to report an increasing outlet flow rate, around 10:45 where this stabilizes around [redacted] kg/h which is the vapour phase coming originally from the main tower condensers. Simultaneously, the reflux flow rate stabilizes around [redacted] kg/h which is the liquid phase from the main condensers.



[redacted]

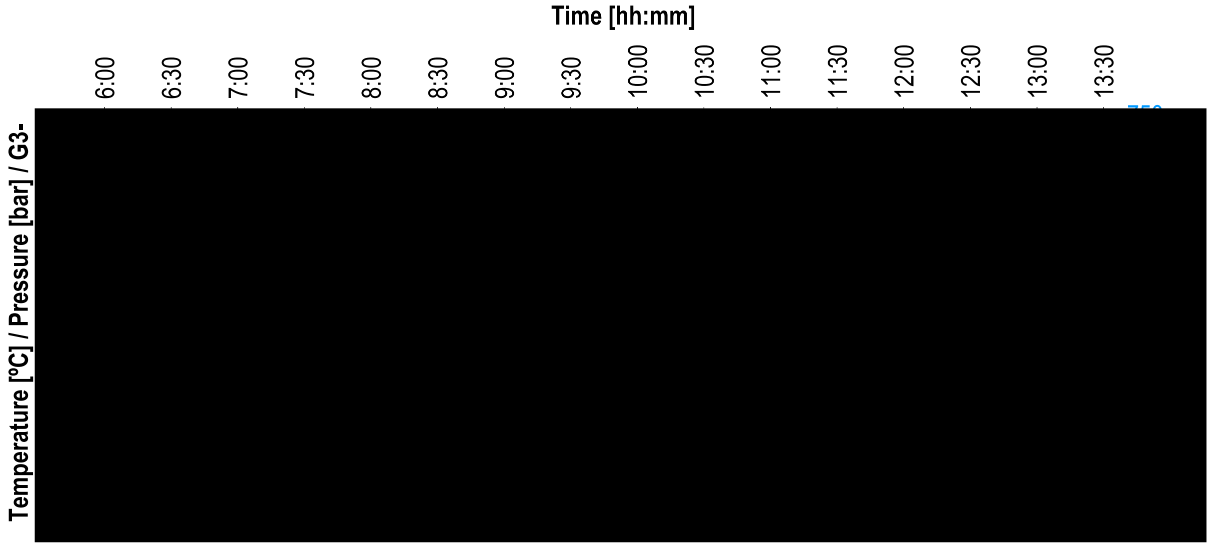
Figure 5.15. Flowrate of liquid phase from reflux drum.

[redacted]

Figure 5.16 . Flowrate of gaseous phase leaving vent column.

[redacted]

Figure 5.17. Instrumentation location



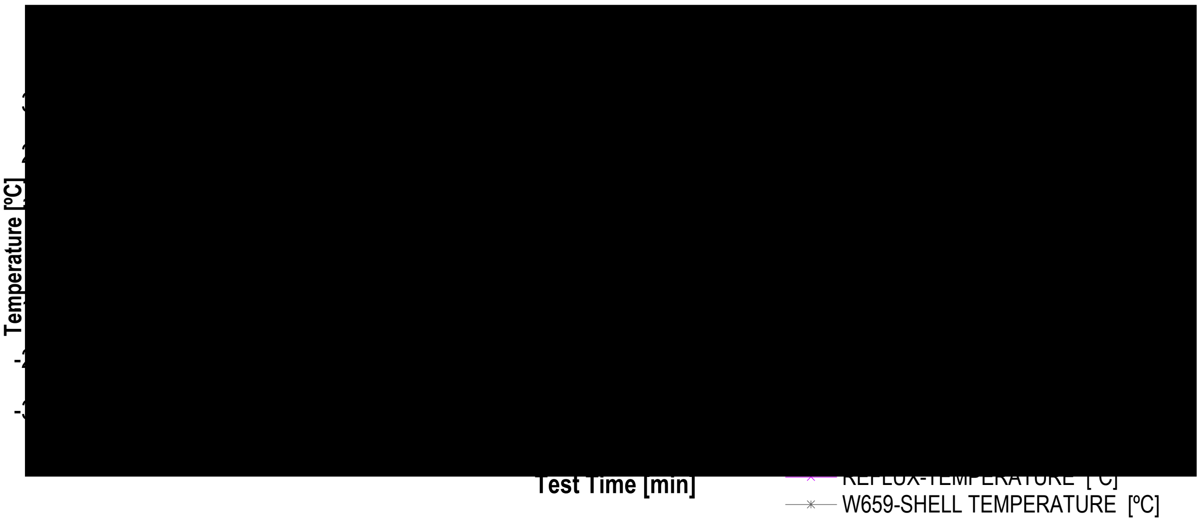
[redacted]

Figure 5.18. Variables time evolution during the stage 3. Microsoft Excel® plots.

[redacted]

Figure 5.19. Variables profile during stage 3. Aspen Process Explorer® plot and table.

Once all the system operate in the new steady state, the last transient state modifying the drum level starts. Close to 11:45 the reflux flow rate is reduced in a 50% (despite the instrumentation reports zero) to avoid drying completely the column trays. At this moment and as the inlet flow rate to the reflux drum remains in the same value, the drum level starts to increase with a constant rate (constant slope) meaning a constant inlet flow rate until around 13:00 when the drum level reaches the maximum value of [redacted]%.



[redacted]

Figure 5.20. G2 and G3 outlet streams temperature time evolution.

Figure 5.21 and Figure 5.22 shows the instant real value of G2 and G3 respectively, before reducing the reflux flow rate. Green lines represent transient state that are not considered as part of the stationary state and therefore these points are not included in the average calculation.

Figure 5.23 shows the level variation during the transient state, from [redacted]%. Green lines are points that does not belong to the transient state of interest. Blue line is the points that belongs to the transient state that identifies the stage 3.

Time [hh:mm]

Flow rate [kg/h]

[redacted]

Figure 5.21. Vent gas flow rate average value.

Time [hh:mm]

Flow rate [kg/h]

[redacted]

Figure 5.22. Reflux flow rate average value.

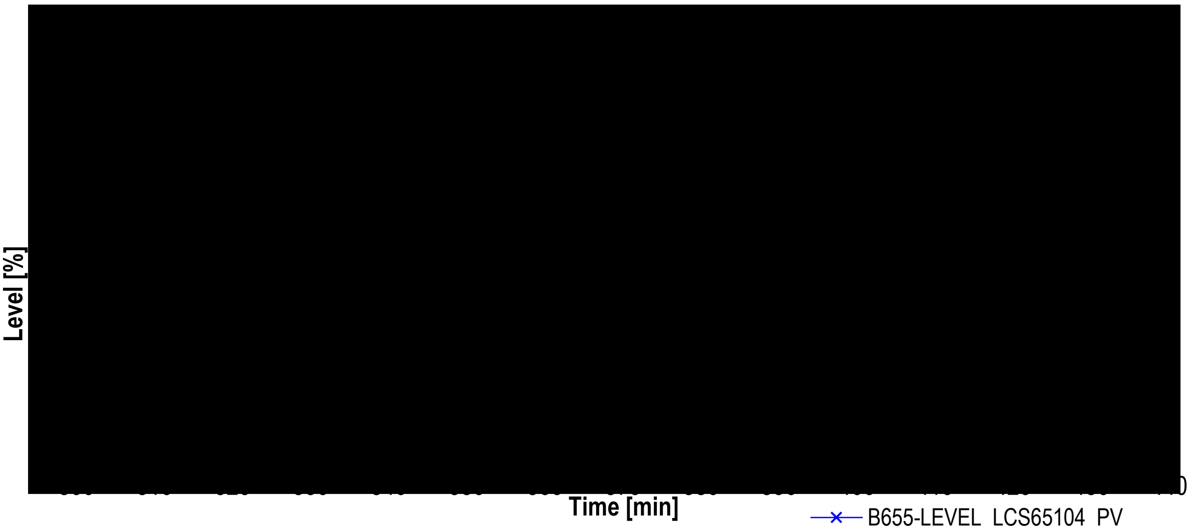

[redacted]

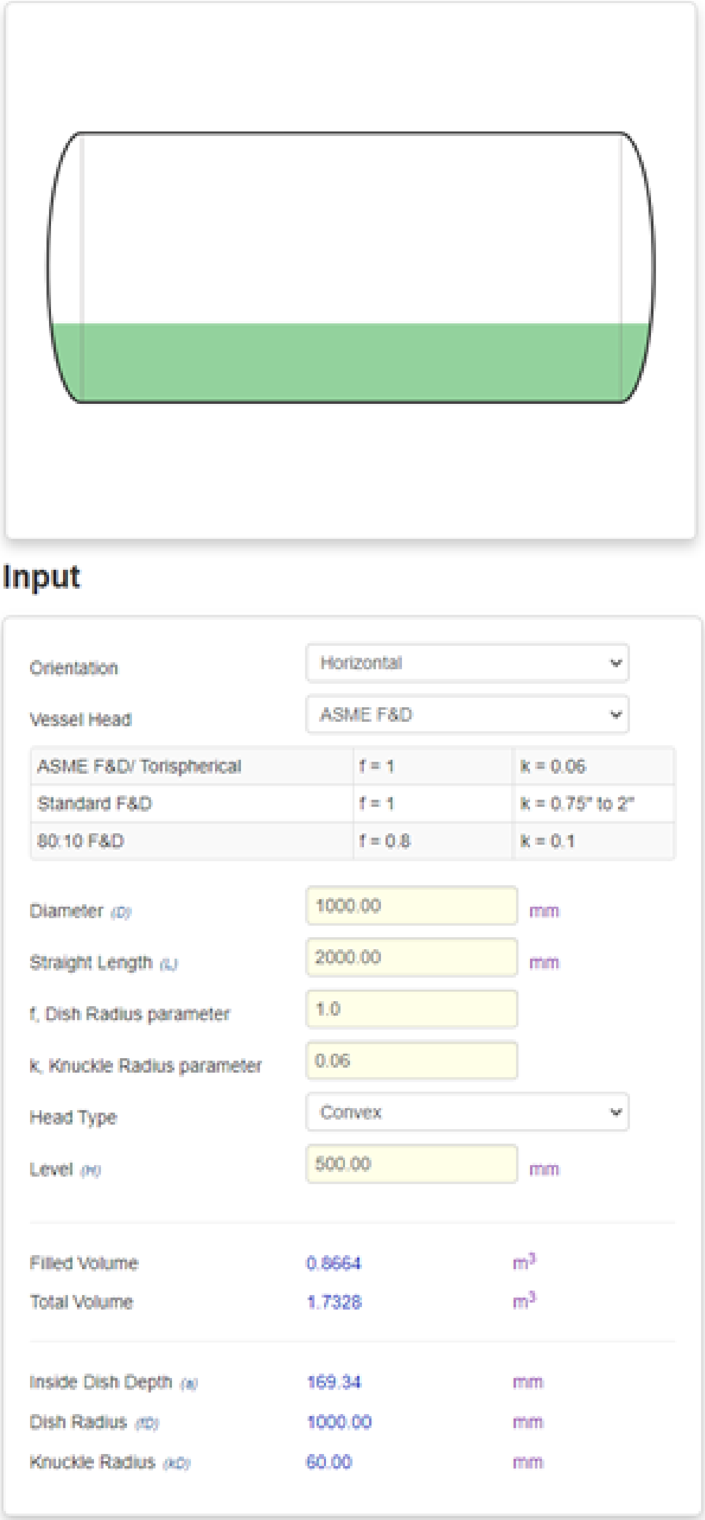
Figure 5.23. Level variation in the transient state in stage 3.

In the Figure 5.23 the boundary conditions of the transient state can be checked. The calculation sheet in Figure 5.24 stablish the volume of an horizontal tank with torispherical heads as a function of the height. Using the Equation (4.11 the inlet flow rate was calculated and the results are summarized in Table 5.43. According to this method, the liquid phase coming from the main tower condensers has a flow rate of [redacted] kg/h.

Table 5.44 summarize all the results obtained to establish the vapour fraction in the real plant. The values estimated by each method are of the same magnitude order with a vapour fraction that goes from [redacted] to [redacted]% compared with the design value of [redacted]%, meaning that effectively the main condensers exchange more heat than the required by design.

Table 5.43. Stage 3 results summary

	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]			
[redacted]	[redacted]	[redacted]	[redacted]	
[redacted]	[redacted]			



Input

Orientation	Horizontal	
Vessel Head	ASME F&D	

ASME F&D/ Torispherical	f = 1	k = 0.06
Standard F&D	f = 1	k = 0.75" to 2"
80:10 F&D	f = 0.8	k = 0.1

Diameter (D)	1000.00	mm
Straight Length (L)	2000.00	mm
f, Dish Radius parameter	1.0	
k, Knuckle Radius parameter	0.06	
Head Type	Convex	
Level (H)	500.00	mm
Filled Volume	0.8664	m^3
Total Volume	1.7328	m^3
Inside Dish Depth (a)	169.34	mm
Dish Radius (fD)	1000.00	mm
Knuckle Radius (kD)	60.00	mm

Figure 5.24. Volume calculation sheet for horizontal tanks with torispherical head.

5.2.5. Composition estimation of gas phase from W392

The carrier gas phase separation was modelled using the flowsheet defined in Figure 5.25. Assuming both phases are in equilibrium, for specific temperature and pressure operating conditions and composition of the liquid phase, the vapour phase composition is only one and is fully determined by the liquid-vapour equilibrium.

Table 5.44. Results summary of vapour fraction tests

[redacted]	[redacted]	[redacted]			[redacted]			[redacted]
		[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

[redacted]

Figure 5.25. Carrier gas phase separation in heat exchanger W392.

W392 operating pressure is measured ([redacted] bara) but the temperature it is not. Then, using the Sensitivity module (Figure 5.26) in the Model Analysis Tools, the system temperature was manipulated from [redacted] and [redacted]ºC to find the liquid phase with the same composition reported in Table 5.38. These results can be seen in Figure 5.27. This temperature is loaded in the input of [redacted] and the mass fractions obtained in the stream [redacted] is the composition of the vapour phase and are introduced in Table 5.45.

[redacted]

Figure 5.26. Sensitivity analysis input from Aspen Plus® v.12.

[redacted]

Figure 5.27. Temperature results from the sensitivity analysis.

Table 5.45. [redacted] gas phase mass fractions.

[redacted]	
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]

The system propane/propylene is well known by the small difference in relative volatility that make this system a big challenge to separate. A side finding from this simulation is shown in Figure 5.28, according to Aspen Plus® results the liquid stream can convert into a vapour stream just by heating from 43,20ºC to 43,95. A control loop to control this temperature would be a true challenge considering the accuracy in the temperature measurement made by a thermocouple (Table 5.46).

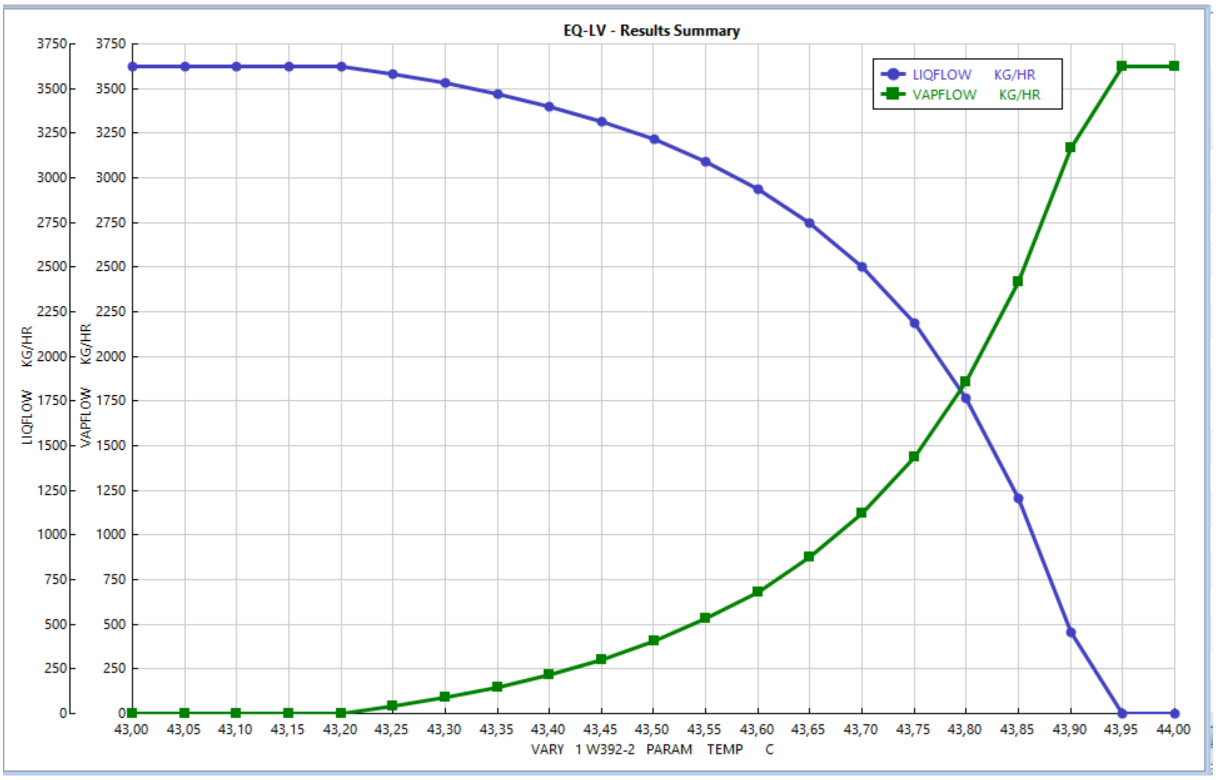


Figure 5.28. Vapour mass flowrate increasing the temperature.

Table 5.46. Thermocouple accuracy by type and temperature. (Source: www.ni.com)

Temperature [ºC]	Type							
	B	E	J	K	N	R	S	T
-200	-	-	-	3.0	3.0	-	-	3.0
-100	-	-	-	2.5	2.5	-	-	1.5
0	-	1.7	1.5	1.5	1.5	1.0	1.0	0.5
200	-	1.7	1.5	1.5	1.5	1.0	1.0	0.8
400	-	2.0	1.6	1.6	1.6	1.0	1.0	-
600	1.5	3.0	2.4	2.4	2.4	1.0	1.0	-
800	2.0	4.0	-	3.2	3.2	1.0	1.0	-
1000	2.5	-	-	4.0	4.0	1.0	1.0	-
1200	3.0	-	-	9.0	9.0	1.3	1.3	-
1400	3.5	-	-	-	-	1.9	1.9	-
1600	4.0	-	-	-	-	2.5	2.5	-

5.2.6. PFD real plant version

All the information compiled in Section 4.2 define the PFD real plant version showed in Table 5.47. To the right of the table an additional column is added where an overall mass balance is performed to check consistency between inlets and outlets. The global mass balance of mass flows reports a difference of 40,47 kg/h. Comparing this with inlets and outlets total mass flows represents around a [redacted]% of error and can be easily associated with the instrumentation measuring error despite the effort made to be reduce the error of the methodology applied in this project.

It is important to highlight that propylene outlet is higher than the feed inlet. This produce a recovery higher than 100% which is not possible to accomplish. The deviation can also be associated with the product and feed flow rate measurement.

The top vapour stream composition and flow rate was no possible to obtain but the basic operating conditions were measured and included in the PFD final version.

One last very important parameter obtained is the global efficiency. With the feed stream fully defined in

Table 5.48, the shortcut ditillation module from Aspen Plus® is used to establish the theoretical number of stages to produce the same distilled stream. The input loaded is sumarised in Table 5.49. The recoveries are again calculated with feed stream and product (distillate) flow rates and compositions. The reflux ratio is a controlled variable and was taken from the DCS records in the period analysed. The results are introduced in Table 5.50 reporting a real Global Efficiency around the [redacted]%.

5.3. Comparing design basic engineering and real plant operation

The feed stream flow rate comparison is introduced in Table 5.51. The real plant feed flow rate is higher than the expected by design processing almost twice the flow it was designed for.

The composition estimations for the case of the uncondensed feed stream (gas phase from [redacted]) are not the real but are presented to compare the content and check it against the design. As can be seen in Table 5.52, the propylene content is quite similar but propane differs. All others components are present in a lower proportion compared with the design. The liquid phase feed (or condensed) content in propylene is higher and in propane lower. The rest of the component are more distributed but in traces.

The internal streams flow rates comparison (Table 5.54) introduce the main difference in the operation. By design, the optimal operation point is obtained with a reflux rate of around [redacted] kg/h but in the real plant the column operates almost with the [redacted]% of that value, [redacted]kg/h. Higher reflux rate means higher energy consumption but also produce distilled more concentrated in the target component increasing product quality and purity. Top vapour flow rate were not stablished but is similar in magnitude to the reflux leaving the reflux drum.

The reflux mass fractions are reported in Table 5.55. The internal streams content is slightly different but is composed by almost the same components except for hydrogen and nitrogen. Methane and ethane content is not the same for both as it was conceived in the design.

Table 5.47. Process Flow Diagram Data. Real plant version.

[redacted]

Table 5.48. Real plant feed flow rates, composition, and properties.

[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]

[redacted]	[redacted]
[redacted]	[redacted]

Table 5.49. Shortcut distillation input. Real plant data.

Input	Value
Column specifications - Reflux ratio	[redacted]
Pressure - Condenser [bara]	[redacted]
Pressure - Reboiler [bara]	[redacted]
Key components recovery in distillate - LK	[redacted]
Key components recovery in distillate - HK	[redacted]

Table 5.50. Shortcut distillation results for real plant data.

Variable/parameter description	Value
Minimum reflux ratio	[redacted]
Actual reflux ratio	[redacted]
Minimum number of stages	[redacted]
Number of actual stages (theoretical)	[redacted]
Feed stage	[redacted]
Number of actual stages above feed	[redacted]
Reboiler heating required [kW]	[redacted]
Condenser cooling required [kW]	[redacted]
Distillate temperature [ºC]	[redacted]
Bottom temperature [ºC]	[redacted]
Distillate to feed fraction	[redacted]

Table 5.51. Feed streams flow rates comparison.

	Mass flows [kg/h]	
Stream	**Design**	**Process Data**
Uncondensed	[redacted]	[redacted]
Condensed	[redacted]	[redacted]
Total feed	[redacted]	[redacted]

Focusing on the external streams, Table 5.56 and Table 5.57 board the comparison of flow rates and mass fractions respectively. Flow rates are quite different in both scenarios. During the sampling period, no vent gas was reported by the flow meter. The product flow rate extracted is more than twice the design value and the bottom stream flow rate is lower. It is possible to identify that heavier compounds are present in the bottom stream and the lighter can be found in the top stream. The product propylene content is lower than the imposed in the design with a bit lower purity due to a propane higher amount. In general, all the components are present in a similar amount compared with the design. The bottom is composed by no heavy hydrocarbons and by more propane that in the design.

Table 5.52. Uncondensed feed stream compositions comparison.

Stream	**Components**	**Mass fractions**		***Relative error [%]***
		Design	**Process Data**	
Uncondensed	[redacted]	[redacted]	[redacted]	*(63,13%)*
	[redacted]	[redacted]	[redacted]	*(72,60%)*
	[redacted]	[redacted]	[redacted]	*(92,32%)*
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	*(0,62%)*
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	*(100,00%)*
	[redacted]	[redacted]	[redacted]	*(100,00%)*

Considering other specifications and operation variables introduced in Table 5.58, the real plant energy consumption is lower which can be explained due to the lower reflux rate in the main tower. In general, all the utilities consumption are lower in the real plant than in the design. The top vapour temperature, the reflux temperature and the bottoms temperature reports a lower value. The propylene mass recovery in the side stream product is higher than the [redacted]% which is not possible and must have included the error introduced by the methodology applied to characterise the real plant data and the instrumentation as well. The propylene mass fraction in bottom is higher in the real plant that in the design but it is important to have in mind that the flow rate is much lower in the real plant than in the design. The vapour fraction in the real plant is lower and can be assigned to higher energy consumption compared with the designed condenser performance. No vent gas flow rate was reported meaning no propylene losses was registered.

The vent column reflux is the liquid phase that comes from the vent condenser and ends in the reflux drum to then go back as reflux to the main tower. If the vent condenser is operating normally no vent gas leaves the vent column. If the condenser does not operates the vent gas stream is more than zero and was estimated around [redacted]kg/h in the vapour fraction test section (5.2.4). This reflux represents the less than the 10% of the defined in the original design. A similar analysis can be made to estimate the vent column tray 1 vapour and liquid flow rates.

Table 5.53. Condensed feed stream compositions comparison.

Stream	**Components**	***Mass fractions***		***Relative error [%]***
		Design	**Process Data**	
Condensed	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	*(47,85%)*
	[redacted]	[redacted]	[redacted]	*(16,94%)*
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	*(99,99%)*
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-

Table 5.54. Internal streams. Flow rates comparison.

Stream	**Mass flows [kg/h]**		
	Design	**Process Data**	**Relative error [%]**
Top Vapour	[redacted]	[redacted]	[redacted]
Reflux	[redacted]	[redacted]	[redacted]

Table 5.55. Internal stream. Compositions comparison.

Stream	**Components**	**Mass fractions**		**Relative error [%]**
		Design	**Process Data**	
Reflux	[redacted]	[redacted]	[redacted]	*(99,21%)*
	[redacted]	[redacted]	[redacted]	*(254,07%)*
	[redacted]	[redacted]	[redacted]	*(21,00%)*
	[redacted]	[redacted]	[redacted]	*(37,55%)*
	[redacted]	[redacted]	[redacted]	*(1,43%)*
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	*(100,00%)*
	[redacted]	[redacted]	[redacted]	*(100,00%)*

Table 5.56. External streams. Flow rates comparison.

Stream	**Mass flows [kg/h]**		
	Design	**Process Data**	**Relative error [%]**
[redacted]	[redacted]	[redacted]	(118,62%)
[redacted]	[redacted]	[redacted]	(100,00%)
[redacted]	[redacted]	[redacted]	(70,04%)

Table 5.57. External streams. Mass fractions comparison.

Stream	**Components**	**Mass fractions**		**Relative error [%]**
		Design	**Process Data**	
Product	[redacted]	[redacted]	[redacted]	(100,00%)
	[redacted]	[redacted]	[redacted]	(36,71%)
	[redacted]	[redacted]	[redacted]	(35,01%)
	[redacted]	[redacted]	[redacted]	(39,08%)
	[redacted]	[redacted]	[redacted]	(1,05%)
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
Bottom	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	(86,40%)
	[redacted]	[redacted]	[redacted]	(95,39%)
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	(99,85%)
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-

Table 5.58. Other specifications and operation variables comparison.

Variable	***Units***	***Design***	**Process Data**	**Relative error [%]**
[redacted]	[redacted]	[redacted]	[redacted]	*(81,60%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(51,58%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(72,62%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(5,81%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(22,73%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(23,36%)*
[redacted]		[redacted]	[redacted]	-
[redacted]		[redacted]	[redacted]	*(79,42%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(4,12%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(95,39%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(63,62%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(100,00%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(100,00%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(91,10%)*
[redacted]	[redacted]	[redacted]	[redacted]	-
[redacted]	[redacted]	[redacted]	[redacted]	-
[redacted]	[redacted]	[redacted]	[redacted]	-
[redacted]	[redacted]	[redacted]	[redacted]	

Comparing global efficiencies, the real plant show the highest. The design feed stream has a lower propylene content including C4 heavy HC compared to the actual feed stream that has traces of heavy HC. This is attributed to the fresh propylene polymer grade that is used nowadays (Table 5.59). Other factor is the recovery imposed to the shortcut distillation for both cases. The better the separation of components in the distillation, the higher the purity in LK and HK components in distilled and bottom stream respectively, the greater the number of theoretical stages required and the greater the column global efficiency compared with the actual number of trays installed in plant.

Table 5.59. Polymer grade propylene supplied by the provider.

01/09/2022-29/09/2022				
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

The temperature profile for both cases is reported in Figure 5.29. From the tray Nº20 to Nº95 the real plant follows a profile very similar to the design. In bottoms, the design temperature include heavy hydrocarbons and is expected that the temperature is higher than without them. In top section, the decreasing temperature can be assigned to the reflux temperature that is quite lower that the top vapour temperature in real plant data.

Temperature [ºC]

[redacted]

Figure 5.29. Distillation temperature profile comparison.

5.4. Model calibration with real plant data

The model calibration is made by updating the inputs of the base case (Figure 5.8) with the data defined in Table 5.47 that are inputs required to build the simulation. In addition to inputs as flow rates, compositions and operating conditions, the column is also updated with the global efficiency obtained for real plant conditions as shown in Figure 5.30.

[redacted]

Figure 5.30. HYSYS ® efficiency tuning view for the distillation column. Group A and B correspond to enrichment and rectification section respectively.

In general, the model predict the real data very accurate. The internal streams predictions are very close to the observed value (Table 5.60). The reflux compositions are not very accurate but are predicted around the magnitude order observed (Table 5.61). The top vapour flow rate and compositions (Table 5.62) in the real plant has no instrumentation installed for flow measurement at the time of this project but it was interesting to include the predictions to have at least and indications of what this should be.

The external stream flow rates were also well predicted as can be noted in Table 5.63. Despite the error is not low but the product concentration in particular is very accurate compared with the real data (Table 5.64). For the vent gas stream the predictions are also included just to provide an estimation of flow rates and compositions (Table 5.65). Last, for the bottom stream and as the assumption was to neglect the heavy components, these concentration were not predicted by the model. Even though, these are present as traces in the bottom real data (Table 5.66).

Table 5.60. Real plant internal streams flow rates. Model predictions compared with observed values.

	Mass flows [kg/h]		**Relative error [%]**
Stream	**Observed**	**Predicted**	
Top Vapour	[redacted]	[redacted]	-
Reflux	[redacted]	[redacted]	*(0,00%)*

Table 5.61. Real plant reflux mass fractions. Model predictions compared with observed values.

Stream	Components	***Mass fractions***		**Relative error [%]**
		Observed	**Predicted**	
Reflux	[redacted]	[redacted]	[redacted]	*(95,45%)*
	[redacted]	[redacted]	[redacted]	*(98,39%)*
	[redacted]	[redacted]	[redacted]	*(99,79%)*
	[redacted]	[redacted]	[redacted]	*(58,64%)*
	[redacted]	[redacted]	[redacted]	*(1,31%)*
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-

Table 5.62. Real plant top vapour mass fractions. Model predictions compared with observed values.

Stream	**Component**	**Mass fractions**		**Relative error [%]**
		Observed	**Predicted**	
Top vapour	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-

Table 5.63. Real plant external streams flow rates. Model predictions compared with observed values.

Stream	**Flow rates [kg/h]**		**Relative error [%]**
	OBSERVED	**PREDICTED**	
Product	[redacted]	[redacted]	*(0,00%)*
Vent Gas	[redacted]	[redacted]	-
Bottom	[redacted]	[redacted]	*(1,28%)*

Table 5.64. Real plant product mass fractions. Model predictions compared with observed values.

Stream	**Components**	***Mass fractions***		**Relative error [%]**
		Observed	**Predicted**	
Product	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	*(14,29%)*
	[redacted]	[redacted]	[redacted]	*(17,30%)*
	[redacted]	[redacted]	[redacted]	*(24,19%)*
	[redacted]	[redacted]	[redacted]	*(0,96%)*
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-

Table 5.65. Real plant vent gas mass fractions. Model predictions compared with observed values.

Stream	**Components**	***Mass fractions***		**Relative error [%]**
		Observed	**Predicted**	
Vent Gas	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-

Table 5.66. Real plant bottom mass fractions. Model predictions compared with observed values.

Stream	**Components**	***Mass fractions***		**Relative error [%]**
		Observed	**Predicted**	
Bottom	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	*(1,30%)*
	[redacted]	[redacted]	[redacted]	*(1,30%)*
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	*(100,00%)*
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-
	[redacted]	[redacted]	[redacted]	-

In Table 5.67 the other operation variables and column specifications are reported. The duties required by the operation were estimated similar to the real data except for the main condenser. Here, the predictions are higher than calculations for the real data. The bottom temperature show again a considerable deviation in the mixture temperature. In the real process, bottoms include other heavy components as water, oil and others that accumulates in that section increasing the temperature. The model is not capable to take this under consideration. The reflux temperature also introduce deviation that can justified in a small portion by the environment temperature in December. The reflux leaves the reflux drum and is conducted to pumps that sent this to the top of the column. During this path a temperature drop is experienced by exchanging heat with the surrounding in the real plant decreasing the reflux temperature. For the rest of the variables considered the predictions includes error less than 5% meaning accurate results.

The model adjustment evaluation was made with the MAI used in the previous sections and are reported in (Table 5.68). These value indicates that a global average error is around 46%.

The temperature profile along the column in included and compared with the real plant data. Both in top and bottom are observed deviations related with the assumptions made mentioned in the previous paragraph: absence of heavy compounds in bottoms and a minor heat exchange with the surroundings.

Table 5.67. Real plant specifications and operation variables.

Variable	***Units***	***Observed***	**Predicted**	**Relative error [%]**
[redacted]	[redacted]	[redacted]	[redacted]	*(145,71%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(6,41%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(58,33%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(10,78%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(34,28%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(54,93%)*
[redacted]		[redacted]	[redacted]	-
[redacted]		[redacted]	[redacted]	*(0,00%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(1,33%)*
[redacted]	[redacted]	[redacted]	[redacted]	*(1,30%)*
[redacted]	[redacted]	[redacted]	[redacted]	-
[redacted]	[redacted]	[redacted]	[redacted]	-
[redacted]	[redacted]	[redacted]	[redacted]	-
[redacted]	[redacted]	[redacted]	[redacted]	-
[redacted]	[redacted]	[redacted]	[redacted]	-
[redacted]	[redacted]	[redacted]	[redacted]	-
[redacted]	[redacted]	[redacted]	[redacted]	-

Last, with the model calibrated it is possible to obtain the composition profile. Based on this and on the feed stream composition the optimal feed tray location can be stablished. As the feed stream composition changed compared wih the design, the optimal tray or stage changed as well. Figure 5.31 show the composition profile and define the optimal feed tray location. For the feed stream composition from Table 5.47 the location corresponds to the tray Nº [redacted].

Table 5.68. Model adjustment parameters.

Parameter	Value
Model Adjustment Index (MAI) – Mean square relative error	[redacted]
Root mean square relative error	[redacted]

Temperature [ºC]

[redacted]

Mass fraction [%w/w]

[redacted]

Figure 5.31. Composition profile and optimal feed stage location. Real plant feed stream.

5.5. Improvement & Optimization

5.5.1. Reflux rate effect

5.5.1.1. Theoretical

Figure 5.32 introduce the results of a case of study performed using the base case model with feed stream B and PR as fluid package. The minimum reflux rate of [redacted] kg/h is obtained using the minimum reflux ratio from the shortcut distillation results in Table 5.5 and the distilled mass flow rate. The dashed line in [redacted]% is the minimum mass recovery imposed by the recovery section design.

As can be seen in the plot, for higher reflux rate the propylene mass recovery increase while increasing the energy consumption. For higher values than [redacted]kg/h no significant increase is observed in the distilled propylene mass recovery. For lower reflux rates than around [redacted]kg/h the minimum [redacted]% of propylene mass recovery is not accomplished. Therefore, the suggested operation window goes from reflux rates of [redacted] to [redacted]kg/h.

No significant changes are observed in the vent column condenser heat flow. The amount of propylene that escapes with the NCG and light hydrocarbons leaving the reflux drum is increased for higher reflux rates but it is not meaningful remaining approximately constant.

[redacted]

Figure 5.32. Reflux rate effect in propylene mass recovery in distillate and utilities consumption.

5.5.1.2. Empirical

Focusing on the sources of propylene and propane introduced in the [redacted] reactor, Figure 5.33 define the amount of each of these components using a black box approach. [redacted] propylene composition comes from the data reported by the provider in September of 2022. Distilled and Carrier Gas streams propylene and propane content comes from the composition analysis. Comparing fresh propylene with the distilled concentration, the major amount of propane introduced in the reaction atmosphere comes from the last. Therefore, improving the distillation performance has the higher impact in the reaction rate.

[redacted]

Figure 5.33. Propane and propylene reactor inlet and outlets.

The first phase test summary information is presented in Table 5.69 adding the types of product produced in the tested period. The main objective of this preliminary test was to evaluate the reboiler purge system response which was normal and then prove or disprove the Figure 5.36 show the Aspen Process Explorer® trend module during the test including the time evolution of the selected variables and Table 5.70 summarize the average values of the analysed variables. At first sight. no abrupt changes is observed in the variables trend meaning that no outer causes affect the system response and the results obtained can be assigned only to reflux ratio changes. The propane content decrease by increasing the reflux ratio. The bottom stream flow rate is practically constant for both cases. The bottom temperature increase and can be explained to a higher propane content in bottoms increasing the bubble point temperature of this stage. Something similar happens to the product stream, the temperature tends to the pure component boiling point (Table 5.70).

The energy consumption increase for the case of the low pressure steam. Higher reflux ratio implies that for a fixed side product flow rate, a higher reflux rate is sent back to the column and at the bottoms a higher amount of liquid needs to be evaporated to become a vapour phase. Cooling water and propylene flow rate does not vary and remains in the same value.

Table 5.69. 1st Phase test summary.

Reflux ratio	Start date	End date	Length [h]	Product
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]

Table 5.70. Reflux rate effect results. 1st Phase empirical test.

Variable description	**Reflux ratio**	
	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]

TBP – Propane [ºC] 49,678

TBP – Propylene [ºC] 41,401

[redacted]

Figure 5.34. Analysed variables time evolution during the first test. (a) Reflux ratio of 1.4.

[redacted]

Figure 5.36. Analysed variables time evolution during the first test. (b) Reflux ratio of 1,8.

The second phase test summary information is compiled in Table 5.71. A wider interval of reflux ratio values is analysed in this test and the maximum reached was higher than in the preliminary test.

Table 5.71. 2nd Phase test summary.

Reflux ratio R	Start date	End date	Length [h]	Product
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	

During the course of this test in the distillation section another test in the reaction section was performed. The last, was to try a new type of catalyst called [redacted]®, part of the [redacted]® Technology. To operates the [redacted]® process installed with the new catalyst, some changes must be introduced in the equipment usual operation. This introduces external disturbances that affected the results observed in the reflux tests but should not be interpreted as a consequence of the latter. In the distillation section, one of the modifications introduced was to switch the distillate destination. In the usual operation the distillate goes to the [redacted] propylene storage tank but for the new catalyser the distillate is sent directly to the [redacted] reactor. In Figure 5.37 can be identified when this perturbation is introduced. The distillate sent to the [redacted] tank was sent to the reactor the 09/02/2023 at 18:39:09. This is represented in the figure with the vertical dashed line in red. Therefore, the analysis of the results obtained in the reflux rate test takes under consideration additional perturbations this may introduce and how this affects the analysed variables. Operating conditions to the left of the dashed line was defined as conditions A and the other as conditions B. It is not correct to analyse the effect produced mixing both conditions A and B, it was analysed separately.

Time [dd/mm hh:mm]

[redacted]

Figure 5.37. New catalyser test start time.

Figure 5.38 introduces the propane mole fraction time evolution for the reflux ratio values analysed. It can be noticed that in the first portion of the plot the propylene decrease as the reflux ratio increase. Then, the modifications introduced to use [redacted]® modifying conditions from A to B produces a significant increase in the propane concentration. Finally, the propane concentration tends to decrease and stabilize in a new steady state for conditions B. For fixed operating conditions, increasing the reflux rate decrease the propane concentration in the reaction atmosphere inside the reactor. Despite the error reported in the chromatography analysers calibration (Table 4.3) the propylene trend is also introduced in Figure 5.38, but only for performance checking purposes. The values measured does not show the theoretical expected response.

Other alternative to check the purity of the product stream is the distilled temperature. Based on the compositions reported in Table 5.38 the distilled can be easily considered as propane/propylene binary mixture. If the propylene purity increase the tray Nº[redacted] temperature will be closer to the pure propylene TBP at the operating pressure. Figure 5.39 introduce the tray 95 temperature time evolution compared with the propylene TBP. Considering the thermocouples accuracy, increasing the reflux ratio produce that the tray 95 temperature tends to the pure propylene TBP. This also prove the theoretical predictions made by the model.

Time [dd/mm hh:mm]

Propane mole fraction/Reflux ratio

[redacted]

Propylene mole fraction

Figure 5.38. Propane and propylene mole fraction time evolution.

Time [dd/mm hh:mm]

Reflux ratio

[redacted]

Temperature [°C]

Figure 5.39. Distillate product temperature time evolution.

Figure 5.40 introduces the low pressure steam, cooling water and refrigerant propylene flow rates to estimate flow rate effect over the utilities consumption. Both cooling water and refrigerant propylene remain constant and seems to be unaffected by the reflux ratio. The steam consumption in the reboiler varies with the reflux ratio increase. In the last twelve hours decrease but this is caused by a feed stream flow rate decrease that can be observed in Figure 5.41. The feed streams to the distillation section decreased, this produces a decrease in the reboiler and the reflux drum level. The control loop sense this and reduce the distillate flow rate (Figure 5.37) to maintain a fixed reflux ratio. Figure 5.41 also includes the bottom stream flow rate which seems to be invariable.

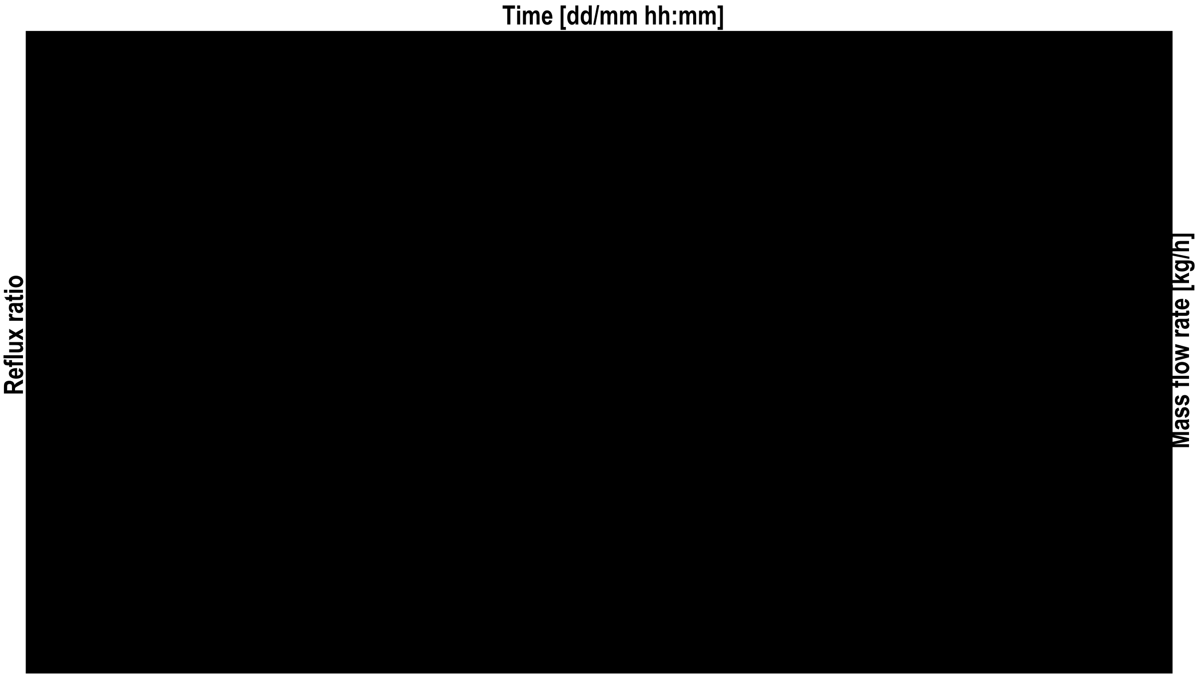


Figure 5.40. Utilities consumption time evolution.

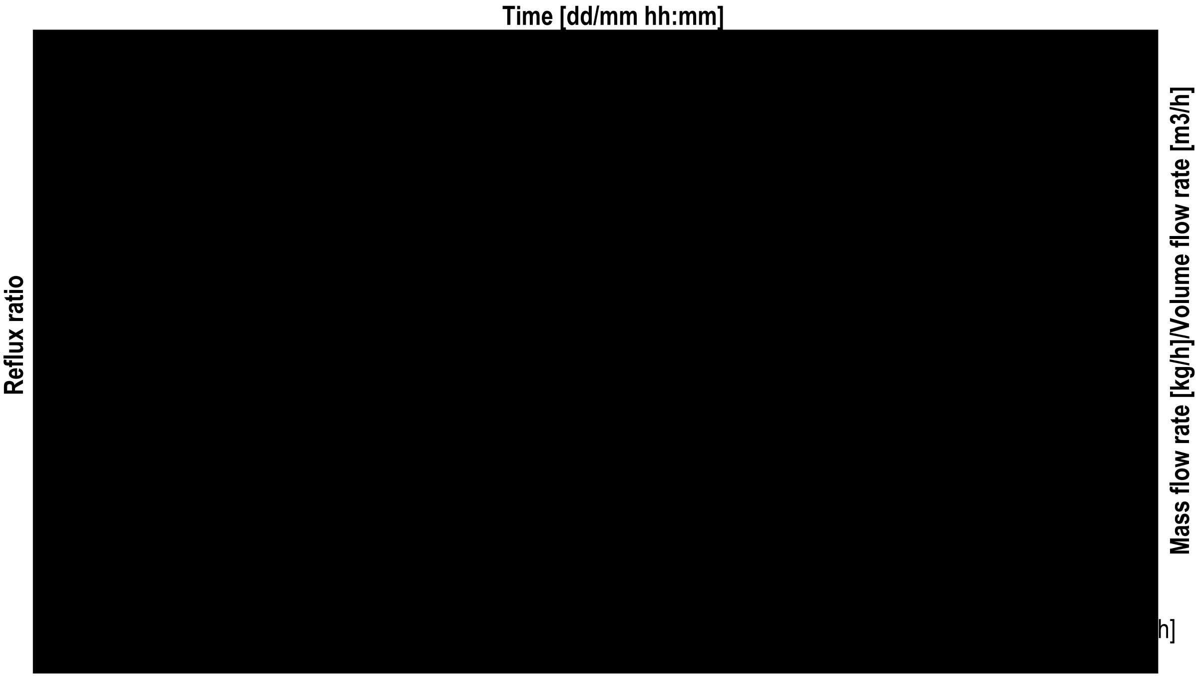


Figure 5.41. Feed and bottom streams flow rate time evolution.

Table 5.72 summarize the average values of all the variables analysed in this section for the tested reflux ratios:

- ✓ The propane mole fraction actually decrease. For the conditions A the concentration goes from [redacted] to [redacted]m/m and from [redacted] to [redacted]%m/m for conditions B. Propane concentration maximum and minimum values present a similar trend except the minimums for conditions B. Propylene mole fraction is included but just to check the response reported by the instrument. The tray Nº95 temperature also tends to the pure propylene TBP (41.401ºC) proving the increasing purity in distillate stream for higher reflux ratios.
- ✓ Increasing the reflux ratio also increase the utilities consumption. Cooling water and refrigerant propylene are almost constant, but the steam flow rate has a sustained increase for higher reflux ratios. The steam consumption goes from [redacted]kg/h for a reflux ratio of [redacted], to [redacted] kg/h for [redacted].
- ✓ The distilled decrease and can be noticed the switch from sending the distillate to [redacted] storage tank to [redacted] reactor. The total flow rate decrease from conditions A to conditions B as a part of adapting [redacted]® installations to use [redacted]® catalyser.
- ✓ Bottom stream shows a minor increase indicating a slightly higher amount of propane leaving the column.
- ✓ Feed streams flow rate are higher for conditions A (total: [redacted] kg/h) than for B (total: [redacted]kg/h). This cause the decrease in the distillate flow rate.

Table 5.72. Reflux ratio test Nº2. Summary results.

[redacted]

5.5.2. Energy optimization

The operation point defined in the basic engineering design is an optimal solution. Therefore, the optimizer methodology was preliminary tested with the design conditions to finally apply it to the real plant scenario. Based on the nature of the objective function and primary variables, the selected data model is Original (for further detailed information see Appendix A4. HYSYS® Optimizer). Figure 5.42 shows the values obtained with the solver for the primary variables. Table 5.73 summarize the calculations performed in the optimizer spreadsheet including flows, compositions, duties, prices, cost, and the objective function evaluation. Notice that in the problem statement the feed and bottom stream price were originally included but it was not possible to find a reasonable price to estimate this cost and add it to the utilities cost.

Comparing results, for the basic engineering design, the suggested reflux ratio of 7 for a distillate flow rate of [redacted]kg/h means a reflux rate of [redacted]kg/h. The optimal solution found by the optimizer with all the assumptions made in the model is [redacted] kg/h. Therefore, the methodology seems to produce reasonable results.

[redacted]

Figure 5.42. Optimizer solution report. Notice the reflux rate low and high bound mentioned in Section 4.5.3.

Table 5.73. HYSYS ® optimizer spreadsheet. Basic engineering design data.

[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]		
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]		
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]

[redacted]	[redacted]

For the real plant scenario, the results obtained from the optimizer datasheet is introduced in Table 5.74. Finally, Table 5.75 define the optimal solution converted into operating conditions and specifications. For the real plant inputs (feed flow rate, compositions, etc) the reflux rate found is higher than the one obtained for the design. As seen before, the recovery section is operating with a different feed stream composition, with a higher propylene concentration. Then, to obtain the minimum mass recovery imposed by the design, the column must operate at higher concentrations to obtain almost pure propylene. This distillation operation requires a higher reflux ratio implying higher energy requirements to condensate and evaporate the inner streams flows at top and bottom sections. The duties estimated for the optimum are higher compared to the obtained in Table 5.73.

Table 5.74. HYSYS ® optimizer spreadsheet. Real plant data.

[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]		
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]		
[redacted]	[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]	[redacted]

[redacted]	[redacted]

Table 5.75. Optimum for operating the C3-Splitter.

[redacted]	
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]
Operation specification	
[redacted]	[redacted]
Utilities flow rates	
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]

The reflux ratio suggested is 4.59. The utilities usual consumption for the last two years was obtained from the DCS records using the trend module in the Aspen Process Explorer® (Figure 5.43). The usual steam flow rate observed was from between 0 and [redacted]kg/h with an average of [redacted] kg/h. In the reflux rate effect test Nº2 the reflux ratio was up to [redacted] (Section 4.5.2), consuming around [redacted]kg/h. Then, the requirement of [redacted]kg/h for a reflux rate of [redacted] for a distillate of [redacted]kg/h seems feasible. The cooling water flowing continuously through the condenser is about [redacted]m3/h, twice the amount required by the optimal operation. The propylene availability for refrigeration purposes can be estimated by two approaches. First, the refrigeration circuit is linked to the carrier gas compressor [redacted] and it maximum capacity of [redacted]kg/h provide a limitation. Second, the fluid availability in the usual operating conditions is imposed by the carrier gas flow rate that comes from the reactor. Focusing on the condensed feed stream flow rate (Table 5.51) the availability can be estimated in [redacted]kg/h. If a higher refrigerant propylene flow rate is needed it could be supplied by fresh propylene. Either way, at this point the refrigeration cycle can easily fulfil the requirements for the optimal operation.

The objective function estimates a profit to compare scenarios. It is one simplified alternative to compare operating points based in similar assumptions. The estimated profit by this means do not consider other costs that should include but it is a simple and direct definition to asses and compare alternatives for operating the recovery section. It is important to keep this in mind for reading and quantify the absolute amount of the profit estimated.

[redacted]

Figure 5.43. C3-Splitter utilities consumption during 2021 and 2022. Aspen Process Explorer ® trend module.

6. CONCLUSIONS

The proposed methodology provided a well-founded technical base to analyse the process, build a model and obtain results from the system behaviour compared with the PPII plant operating.

The basic engineering design data demonstrate that the original polymerization process had differences from the actual version. The main difference lies in the feed stream composition. The process originally conceived include [redacted] and some in amounts comparable with the [redacted] content ([redacted]). Today feed stream composition consequence of the fresh polymer grade propylene coming from the provider has [redacted] and [redacted]. The original composition required a specific column sizing to process it and provide operating conditions for the key components' separation. For today's feed stream the column seems to be oversized. In fact, some visits to the control room showed that [redacted] temperature profile was flat meaning that all the mass transfer occur in [redacted].

From the composition analysis results can be observed that the feed stream has a very good quality: [redacted]%w/w of propylene. The concentration increase produced by the distillation process is not high considering that [redacted] trays are involved: the product leaves the column with a [redacted]%w/w. Therefore, the column may not be working properly with an operation cost high for no significant improvement in the propylene purity. At first sight, this might suggest that, for the new feed conditions a proper evaluation if the distillation is needed under these conditions and as it was conceived with a [redacted]-tray operation divided into two distillation columns or if it is oversized for the actual operation.

The instrumentation installed was enough to characterise the real plant operating conditions. These provided an instant photo of the true distillation operation highlighting the deviation from the design. There are still improving opportunities as instal an ATEX chromatography instrumentation associated to the distillation section or to compute in real time utilities duty with a more accurate methodology. Even though, the data analysed by the DCS, and other instrumentation provide almost any information required of any time, as long as it relevant enough to be recorded in the DCS servers.

Along the years, the optimal operation of a plant can be diluted with changes introduced gradually. For the current operating conditions, flow rates higher than the required by the distillation operation are noticed as a consequence of excessive utilities availability. However, besides this finding there is no flow rates adjustment or control loops linked to limit the use of it. [redacted] supply is one example of this. The cooling towers that provide PPII are sized for supplying a certain water flow with a specific temperature drop. Since these operates generating a cooling water surplus, it is used without analyzing the real requirements by the condenser's operation. And even if the optimum is associated with a lower flow rate than the current, decreasing the flow rates will not have a major impact mainly due to the towers operates at the usual capacity.

[redacted] linked to [redacted] compressor operation go in a similar direction. Even if it is necessary to reduce the flow of this utility, the compressor operation will continue, and a consumption decrease will not represent meaningful savings in electrical energy. In addition to this, the compressor electricity consumption is not comparable with the consumption made by extrusion and the efforts invested into this equipment may not translate into considerable reductions.

Steam flow measurement analyzed in the reflux rate effect tests shows a response that might indicate the valve is not working in the designed opening, or the controlled system is unable to control the flow for the current operation point. The response is similar to a typical critically damped system oscillating indefinitely unable to reach a new steady state. This is not observed for the higher flow rates.

The current feed stream and distillation operating conditions and product obtained indicates the column global efficiency is around the [redacted]%, a higher value than for the same variables defined in the design case characterized by around a [redacted]%.

The optimum obtained by the method in a preliminary analysis seems to be feasible. The utilities availability can provide the required flow rates and tested these new operating conditions to prove or disprove the improvement in the distillation performance theoretically predicted.

Feed stream flow rate of about [redacted]kg/h with a [redacted]%w/w of propylene coming from the reactor carrier gas product flow rate of [redacted]kg/h, a vent gas flow rate of [redacted]kg/h and a bottom stream flow rate of [redacted]kg/h, the reflux rate of [redacted]kg/h impose utilities flow requirements of: [redacted]m3/h of cooling water, [redacted]kg/h of low pressure steam and [redacted]kg/h of refrigerant propylene.

7. FURTHER PROPOSALS

The project scope was completed but from these results other interesting actions appeared and are commented in this section. As a complement, future actions related to the topic are suggested to develop.

✓ From this project, estimated operating conditions were obtained under certain assumptions made during the modelling and data acquisition. These new operating conditions modify the actual based on maximizing the distillate purity reducing the energy consumption on a cost-benefit basis. The theoretical approaches are always deviated from the real life due to the assumptions made, therefore it is strongly suggested to test (*Reflux effect and energy consumption empirical test Nº3*) these new operating conditions before implementing the changes permanently. First, it is important to analyse the individual effect over the recovery section equipment evaluating the response for new conditions and check the predicted benefits and results. Once the variables are individually tested it can be increased slowly as a group and check the overall response and results to get to the final steady state. It is preferably to conclude about the effect of the proposed modifications evaluating the distillate composition directly and for this it is needed a gas chromatography properly calibrated and working instrumentation. If this is not possible, the indirect indication can be performed by monitoring the gas phase propane concentration in the reactor or by checking the tray [redacted] temperature.

✓ The distillate purity is an operation specification that provides a direct indication of the column performance. Any deviation from the best operating conditions can be identified by checking the distillate and bottom composition. It is suggested, for improving the column operation to install chromatography analyser for a continuous evaluation of the column operation on time.

✓ During the execution of this project, it was found that the column temperature profiles was not regularly checked, and the column performance was not the best. For this, it is suggested to regularly check the column temperature profile, especially when other types of polymers are manufactured, and these might introduce changes in the distillation feed (carrier gas) conditions.

✓ Utilities flow rates is an indirect indication about the instant energy consumption, but it is not easily to establish in a specific moment the duty associated to the flow rates. Thus, including the duty associated with every to report it in DCS is very helpful. Other option could be include board with the help of data analysis software implemented by the company such as Trend Miner®.

✓ The utilities that exchange latent heat can be estimated more accurately if the fluid heating or cooling fluid outlet conditions are established at the moment. Reboiler outlet stream can be estimated by the outlet temperature measurement. This will allow to establish if the outlet stream is a mixed vapour and liquid phase or a saturated or subcooled liquid phase.

✓ The optimization approach is based on prices. Even in countries or regions stabilized economically, the prices can be modified with the time. Causes for this can be internals and controllable by the organization or externals and beyond of control. To include this potential variation on the optimum obtained, a price sensitivity analysis is suggested. As the optimizer did not provides data about the shadow price associated with the optimal solution the sensitivity analysis has to be made by other methods that due to time limitations was excluded of the project.

✓ In a similar direction, a technical sensitivity analysis based on the carrier gas flow rate and composition is also recommended. This allows to evaluate the effect over optimal operating conditions by upstream perturbations or modifications. Most of the time, changes introduced in upstream or downstream operating conditions generates perturbations several times along a year and sometimes are not considered in daily operation.

✓ The model calibrated with the real plant data allowed to plot the concentration profile showing that due to the feed stream concentration estimated the feed tray should be Nº[redacted]. It would be interesting to analyse in a regular basis the carrier gas concentration in a year time or other meaningful period. This will stablish if it remains stable and the feed piping can be modified or change and how much does this change. Other relevant information could be, if it is possible, to correlate the type of product polymerized with the carrier gas concentration. Based on all this information the feed could be introduced in the stage with the most similar properties. Notice that, once again, a working and properly calibrated gas chromatography instrumentation will be very helpful to study and improve the distillation performance.

✓ The original scope of this project was to adapt the distillation operation to include an Advanced Distilled Technology such as the mentioned at the end of the introduction. A preliminary analysis of the available space in the PPII plant indicated that, besides the technical feasibility, there was no available space to install all the additional equipment required. PPIII C3-splitter is conceived with vapour recompression, and this was proposed for PPII but no additional space is found to do so with the modifications it implies. To overpass this issue, the literature consulted (Ž. Olujić et alI, 2006) compared vapour recompression (VRC) with heat integrated distillation columns for propylene-propane splitter finding that a reduction in the column height is obtained. A hybrid column, containing in total 20 more trays is 20 m shorter than the VRC (Ž. Olujić et alI, 2006). This can be observed in Figure 7.1.

Therefore, it’s a good alternative to consider for the future. Finally, a techno-economic evaluation was performed, and the results obtained shows that these type of technology could become an economically attractive option for new designs but first there are still some challenges related with the increased design complexity.

✓ In some very particular situations, such as an interruption in the supply of steam, the carrier gas that comes out of the [redacted] heat exchanger is sent directly to the reactor. It is true that according to the production supervisors these events lasted short time to evaluate a longer impact produced by absence of distillation i.e., propane extraction or purge. Propane accumulation in the reactor atmosphere decrease the reactor productivity as was concluded in previous tests in the plant. However, it could be interesting to investigate more about this alternative and the benefits and harms produced during the operation in this mode. In case of technical feasibility could also be interesting to quantify the productivity decrease and energy savings obtained by bypassing the C3-splitter for longer periods.

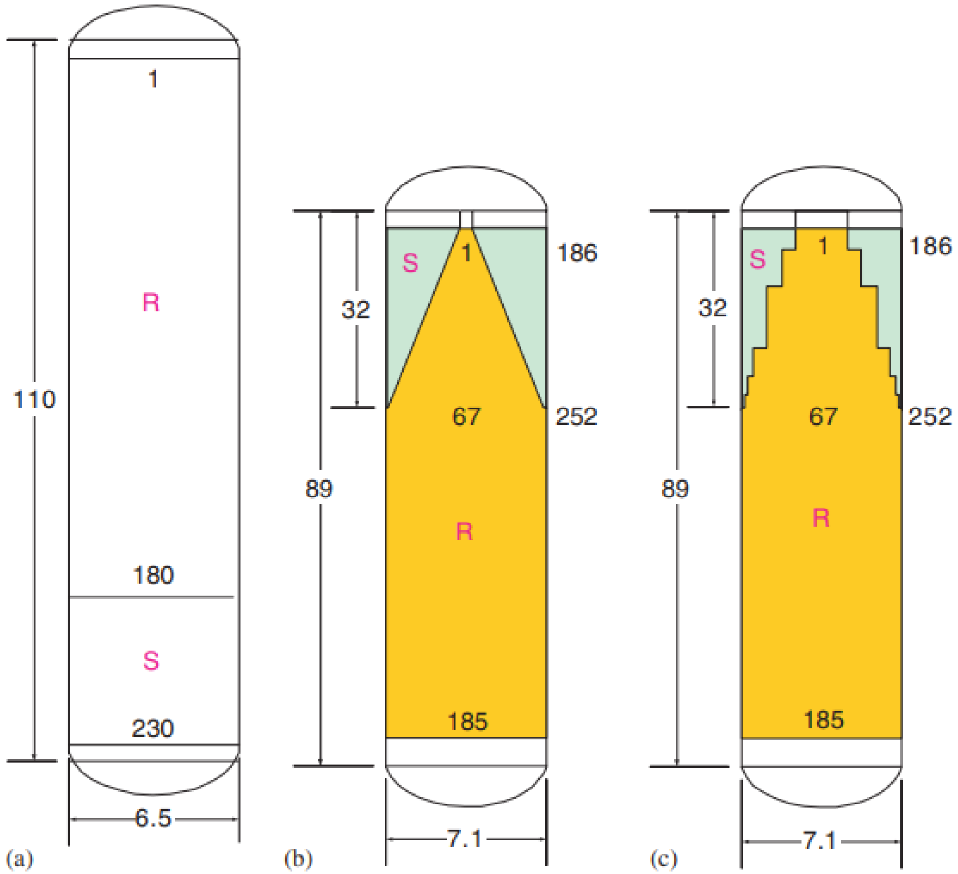


Figure 7.1. Column dimensions of (a) VRC, (b) ideal HIDiC, and (c) a multidiameter HIDiC. Source: Ž. Olujić et all, 2006.

8. REFERENCES

A. S. Foust, L. A. Wenzel, C. W. Clump, L. Maus, L. B. Andersen. Principles of Unit Operations. Wiley India Pvt. 2008.

Anton A. Kiss, Žarko Olujić. A review on process intensification in internally heat-integrated distillation columns. *Chemical Engineering and Processing: Process Intensification*. Dec 2014; 86, 125-144.

Chang Chu En Christopher, Arnab Dutta, Shamsuzzaman Farooq, and Iftekhar A. Karimi. Process Synthesis and Optimization of Propylene/Propane Separation Using Vapor Recompression and Self-Heat Recuperation. *Industrial & Engineering Chemistry Research.* Nov 2017; *56* (49), 14557-14564.

Choudhari A, Divey J. Distillation Optimization By Vapor Recompression. *Chemical Engineering*; Mar 2012; 119(3):43-47.

Hsi-Jen Chen and Yeh-Chin Lin. *Tamkang Journal of Science and Engineering*. Dec 2001; Vol. 4, No. 2, pp. 105-110.

Kazemi, A. et al. (2016) Applied Thermal Engineering, 94, 305-313.

Kiss, A.A. (2013) Advanced Distillation Technology, Wiley.

M. Nakaiwa, K. Huang, A. Endo, T. Ohmori, T. Akiya T. Takamatsu. Internally heat-integrated distillation columns: a review. *Trans IChemE*. Jan 2003; 81, Part A; 162-177.

Tsung-Jen Hoa,, Chi-Tsung Huang, Jhih-Ming Lin, Liang-Sun Lee. Dynamic simulation for internally heat-integrated distillation columns (HIDiC) for propylene–propane system. *Computers and Chemical Engineering*. Jan 2009; 33;1187–1201.

Ž. Olujić, L. Sun, A. de Rijke, P.J. Jansens. Conceptual design of an internally heat integrated propylene-propane splitter. *Energy.* Dec 2009; 31, 15, 3083-3096.

Ž. Olujiæ, L. Sun, M. Gadalla, A. de Rijke, and P. J. Jansensa. Enhancing Thermodynamic Efficiency of Energy Intensive Distillation Columns via Internal Heat Integration. *Chem. Biochem. Eng. Q.* Jul 2008; 22 (4) 383–392.

9. APPENDIX

A1. Basic engineering design data source

[redacted]

Figure 9.1. Carrier Gas Distillation. Process Flow Diagram.

[redacted]

Figure 9.2. Basic Engineering Report

[redacted]

Figure 9.3. Equipment’s Data Sheets

A2. Composition analysis

[redacted]

Figure 9.4. Stream samples extracted for composition determination.

[redacted]

Figure 9.5. Sample extraction installation.

[redacted]

Figure 9.6. Sample extraction point Nº3.

[redacted]

Figure 9.7. . Sample extraction point Nº4.

A3. Real plant process data

[redacted]

Figure 9.8. DCS instant operating conditions during the sample extraction for composition analysis. Carrier gas compression.

[redacted]

Figure 9.9. DCS instant operating conditions during the sample extraction for composition analysis. Carrier gas compression. Propylene destilation I.

[redacted]

Figure 9.10 DCS instant operating conditions during the sample extraction for composition analysis. Carrier gas compression. Propylene destilation II.

[redacted]

Figure 9.11 DCS instant operating conditions during the sample extraction for composition analysis. Carrier gas compression. Reactor R300.

A4. HYSYS® Optimizer

A4.1. Fundamentals

HYSYS contains a multi-variable steady state Optimizer. Once the flowsheet is built and a converged solution has been obtained, the Optimizer can find the operating conditions for minimize (or maximize) a defined Objective Function. The Optimizer is available for steady state calculations only. The operation does not run in Dynamic mode.

The Optimizer possesses a dedicated Spreadsheet to develop the Objective function and any Constraint functions that defines the distillation section operating window. The flexibility of this approach allows you, for example, to construct Objective Functions which maximize profit, minimize utilities or minimize Exchanger UA. This Spreadsheet is identical to the HYSYS Spreadsheet operation; process variables can be attached by dragging and dropping, or using the Variable Navigator

A4.2. Primary variables

These are the variables imported from the flowsheet whose values are manipulated in order to minimize (or maximize) the objective function. Upper and lower bounds are set for all of the primary variables, which are used to define the search range, as well as for normalization.

A4.3. Objective function

The function which is to be minimized or maximized. There is a great deal of flexibility in describing the Objective Function; primary variables can be imported and functions defined within the Optimizer Spreadsheet, which possesses the full capabilities of the main flowsheet spreadsheet.

A4.4. Constraint functions

Inequality and Equality Constraint functions can be defined in the Optimizer Spreadsheet.

Constrained minimization with inequality constraints can be performed using BOX, Mixed, and Sequential Quadratic Programming (SQP) methods.

Problem containing also equality constraints in the statement can be performed with the Original and Hyprotech SQP.

For the case of unconstrained optimization problems, the Fletcher-Reeves and Quasi-Newton methods are available.

A4.5. Data models

Seven data models are available to select, in which the parameters can be set, such as the Optimization Scheme used, the Maximum Number of Iterations, and the Tolerance.

1. Original

2. Hyprotech SQP
3. Selection Optimization
4. DMO
5. LSSQP
6. BOBYQA

The **Original Optimizer** manipulates the values of a set of primary variables in order to minimize (or maximize) a user-defined objective function, constructed from any number of process variables.

$$\min f(x_1, x_2, x_3, ,,,, x_n)$$

where:

$x_1, x_2, x_3, ,,, x_n$ = process variables

In general, the primary variables should not be part of the Objective Function.

Each primary variable, x^0, can be manipulated within a specified range:

$$x^0_{i\ LowerBound} < x^0_i < x^0_{i\ UpperBound} \quad \text{with} \quad i = 1, \ldots, j$$

where:

x_i = a process variable used to define the Objective Function

x^0_i = a primary variable which is manipulated by the Optimizer

y_i = a variable used to define the Constraint Function

The general equality and inequality constraints are:

$$\begin{array}{lcl} c_i(y_1, y_2, y_3, \ldots, y_n) = 0, & & i = 1, \ldots, m_1 \\ c_i(y_1, y_2, y_3, \ldots, y_n) \leq 0, & with & i = m_1 + 1, \ldots, m_2 \\ c_i(y_1, y_2, y_3, \ldots, y_n) \geq 0, & & i = m_2 + 1, \ldots, m \end{array}$$

Constraints are optional and are not supported by all of the Optimization Schemes available in this data model. The BOX, Mixed, and SQP Methods allow for Inequality Constraints. Only the SQP Method incorporates Equality Constraints.)

The **Hyprotech SQP** is a rigorous sequential quadratic programming (SQP) optimization solver. This model can be used to determine the minimum or maximum value of a function of n variables constrained by m inequality and/or equality constraints. You can also impose upper and lower bounds on variables.

The results produced at the end of the optimization run are as follows:

- Values of the Optimizer constraints, variables, and objective function.
- Shadow prices for active constraints.
- A termination reason.
- Iterations and CPU time taken.

Selection Optimization consists of algorithms that solve Mixed Integer Non-Linear Programming (MINLP) problems, in which the objective function is minimized by adjusting both real-valued and binary-valued decision variables.

The algorithms attempt to select a combination of discrete states that a) satisfy the constraints, and b) minimize the objective function. There are two MINLP methods available: Stochastic (also known as the simulated annealing method), and Branch and Bound. These methods use Non-Linear Programming (NLP) optimizers to solve sub-problems.

The **DMO** solver implements a variant of the successive quadratic programming (SQP) algorithm to solve small or large-scale optimization problems. It performs the optimization by solving a sequence of quadratic programming sub-problems.

A5. Utilities

To operate the C3-Splitter three utilities are required:

[redacted]

[redacted]

3) [redacted].

[redacted]

For the case of cooling water and refrigeration propylene, the cost was estimated in terms of the electricity cost.

A5.1. Refrigeration propylene

The unit cost of electricity considered is obtained from the company reports in 2022 (Equation (9.3.1)

[redacted] (9.3.1)

The specific power SP_C is defined as the electrical power required per unit of flow rate provided by the compressor. From the data sheets the total power and flow [redacted] (9.3.2)

[redacted]

[redacted] (9.3.3)

Finally, the cost of the refrigeration in the vent column was estimated with the help of the equation 9.3.4 assuming an annual operating time of the plant of 8000 hours.

[redacted] (9.3.4)

A5.2. Cooling water

Same unit cost of electricity is considered. The specific power required by the [redacted] SP_{RT} is defined in the Equation 9.3.5. In this case, the towers are composed by [redacted].

[redacted]

[redacted] } [redacted]

(9.3.5)

In the condenser de cooling water exchange sensible heat. The flow rate needed is calculated considering the duty (Q_{CI}) required and the enthalpy changes in the water stream (equation 9.3.6). By design, inlet [redacted] (Celsius or Kelvin).

[redacted] ⇒ [redacted] (9.3.6)

Finally, the cost of the cooling in the main condenser was estimated with the help of the Equation 9.3.7.

[redacted] (9.3.7)

A5.3. Low pressure steam

The cost of the steam was estimated considering the flow rate of steam to provide the duty required by the model (Q_R). The unit cost of the steam along 2022 was also extracted from the annual costs reports of that period.

[redacted] (9.3.8)

Once more, according to the reboiler design, steam condensation provide the heat to vaporize the bottoms of the column. Therefore, the required flow rate of steam was estimated with the duty reported by the model and steam latent heat.

Three values of pressure considered in the data sheet of the reboiler without being possible to know which one is the one considered in the final design: [redacted]. For each one of these, latent heat will be slightly different (Tabla X). Using a conservative approach, the highest cost will be obtained considering the higher pressure to estimate steam requirements. Then, the selected pressure was [redacted].

Table 9.1. Steam latent heat as a function of the pressure.

P [bar]	λST [kJ/kg]
[redacted]	[redacted]
[redacted]	[redacted]
[redacted]	[redacted]

[redacted] $\Bigg\} \Longrightarrow$ [redacted] (9.3.9)

Final expression to estimate the cost of the steam is introduced in equation 9.3.10.

[redacted] (9.3.10)