Simulation and optimisation of batch distillation operations

F. Montes [a,b], C. Brand[b], H. Matos[a]

[a] Instituto Superior Técnico, Avenida Rovisco Pais, 1, 1049-001, Lisboa, Portugal
[b] Process Systems Enterprise Limited (PSE), 26-28 Hammersmith Gr, London W6 7HA, United Kingdom

ABSTRACT

Simulation and optimization of distillation operations were carried out using gPROMS ProcessBuilder[®]. A case study was developed for a ternary mixture, using separator models. Two different control methods were analyzed: constant pressure and constant temperature. A batch distillation was carried out in a multi-stage column model. After validation of the results against experimental data, a sensitivity analysis was carried to several operating parameters.

An optimization problem was also carried out. For three different objective functions, the system has improved, by changing different operational constants and having different time control intervals..

KEYWORDS: Batch distillation, Dynamic simulation, Dynamic optimization, Distillation control, gPROMS.

1. INTRODUCTION

Batch distillation has always been an important process in the history of mankind. A huge number of chemical industries rely on batch distillation to separate a multicomponent mixture with only one column, or to distillate a mixture with solid contents [1]. However it is still one of the most complex processes to simulate, control and optimise. Also, the high energy demand and energy wastes are major negative points of this separation method [2].

Many authors dedicated their work in the pursuit of the optimal operating policy for batch distillation column, such as Converse [3] and Robinson [4]. Both compared and analysed the results obtained against the most common operating policies: constant reflux ratio or constant overhead composition. However, most of the early developments made in this area used shortcut models to soften the required computer processing power and the problem complexity. Examples of these shortcuts are the studies realised by authors such as Barolo [5] and Sundaram [6]. With time, nonequilibrium stage models with mass transfer equations from Maxwell-Stefan equation [7] are implemented and modelled for recent studies [8]

Also, different column configurations and their impact in system behaviour were studied. Sorensen [9] and Hasebe [10] studied the inverted column configuration, and compared the results with the performance of a regular column. The implementation of a middle vessel in a batch column has been studied by many authors such as Morari [11] who compared the behaviour of a middle vessel batch column (MVBC) against a regular and an inverted column. On the other hand, Demicoli [12] analysed an azeotropic binary mixture and a zeotropic ternary mixture in a MVBC, showing the possibility of reducing the overall mixture temperature and the start-up time. However, it's the multi-effect batch column system (MEBC) that attains all the present attention: Hasebe and Kurooka [13] compared a MEBC with a continuous distillation system, for different multicomponent mixtures with constant relative volatilities, concluding that the separation performance tends to the same value, for both methods, the greater the number of components in the mixture.

For the industry, an important choice has to be made in terms of optimisation objectives. Time problem optimisations can reduce the amount of recovered products. Optimising the product recovery will increase the operating time, on the other hand. With time, different optimisation strategies appeared, such as the capacity factor objective (CAP) [14],and profit objective functions.

2. MATERIAL AND METHODS

2.1. gPROMS ProcessBuilder[®]

gPROMS[®] ProcessBuilder® v1.0.0 was the platform used for simulation and validation of different batch processes, provided by Process Systems Enterprise ("PSE"). This platform allows the assembly of different flowsheets, using a simple drag and drop system. Another feature of the gPROMS ProcessBuilder® is the optimisation tool, which is able to optimise a continuous or dynamic behaviour of an assembled flowsheet. For that purpose, it is necessary to provide certain unassigned variables which will be optimised. gPROMS[®] NLPSQP solver has been used to optimise the present problems.

2.2. gML[®] Library

The gML[®] library contains steady state and dynamic models for a huge variety of processes. The models are based on mass balances, momentum, enthalpy and many other physical properties and chemical behaviours. The gML[®] library includes the models needed for batch distillation and one stage separators. For the purpose of this work the following models have been used:

- Separator: describes a two-phase flash vessel (liquid-vapour). It is assumed that there is only one liquid phase, and one vapour phase, and that both are at phase equilibrium;
- Tank: This model has the objective to simulate the storage of intermediate/final liquid components / products. The model's dynamics options determine liquid holdup accumulation, after design specifications and geometrical parameters inputs;
- Sink: the end-point of a flowsheet, where a material stream ends/leaves the flowsheet;
- Source Material: beginning point of a material stream into the flowsheet/system, with infinite capacity if wished. The specifications include temperature, component fraction, pressure and/or flows;

- **Controller:** This model describes and simulates the actions of a controller with proportional, integral and derivative gain. The action mode needs to be specified (manual, automatic or cascade model), as well as the type of controller (P, PD, PI or PID);
- **Distillation column:** The distillation column model describes a two-phase (vapour-liquid) distillation column. This column is divided into multiple stages. For each stage there is a mass and energy balance, and by default it assumed that vapour-liquid equilibrium is achieved at each state. Condenser and reboiler sub-models also assume vapour-liquid equilibrium. Different types of reboiler designs and condenser operating policies can be selected (kettle reboiler, thermosiphon reboiler, partial condenser, total condenser…). Depending on the chosen policy, two or three operational specifications are required. These specifications include the reflux ratio, reboil ratio, distillate flowrate, cooling rate, amongst others;
- **Valve:** This model simulates the flow of a fluid through a valve, using mass and energy balances, and flow-pressure drops relations. A dynamic behaviour of the valve stem position may be selected (Dynamic option), varying accordingly to a given position setpoint;
- **Splitter:** The Splitter model divides an inlet stream into multiple outlet streams, depending on the flowrate or split fraction specified;
- **Cooler:** This model simulates a heat exchanger that removes heat from a fluid stream. Three main modes are available: "Mass and energy balances only", "design" and "performance". The first two allow the heat duty exchanged as an input, while "performance" mode is dependent on the performance of the heat exchanger area and transfer coefficient. Only the third operating mode can be used for dynamic behaviour;
- **Pump:** This unit models the behaviour of a fluid through a pump using mass and energy balances.

2.3. Physical properties

gPROMS standard physical property package is Infochem Multiflash ^{TM}. As Multiflash is designed for equation-oriented modelling, it generates analytical partial derivatives and tight convergence of iterations for variables such as temperature, pressure, composition and density. The determination of phase equilibrium is based on the fact that a component's fugacity is equal in all phases, at equilibrium. For a single vapour-liquid system:

$$f_i^V = f_i^L \qquad \text{(eq. 1)}$$

where f_i^V is the fugacity of component i in the gaseous state, and f_i^L is the fugacity of the component i in the liquid state. There are two main categories for the Multiflash fugacity models: equation of state methods and activity coefficients method. With an equation of state method, all thermal properties can be derived from an equation of state. On the other hand, an activity coefficient method derives the vapour phase properties from an equation of state, whereas the liquid

properties are determined from the summation of the pure component properties to which a mixing term or an excess term has been added.

For the present work, the Non Random Two Liquid (NRTL) activity coefficient model was selected.

3. CASE STUDY: SEPARATOR

3.1 – Case study introduction

For the present case study, a flowsheet has been assembled (see figure 1). The initial holdup and the separator dimensions are presented in table 1.

Table 1 - Design and initial molar holdup specifications of the separator model

Component specification		Separator design		
Component	Initial molar holdup (kmol)			
Cyclohexane	30	Vessel heads	Separator height (m)	Diameter (m)
Heptane	40			
Toluene	50	Flat	5	3

The tank re-fill feed has the same composition as Mujtaba [15].

The condenser has its bottom outlet closed at all times, due to the stem position of the respective valve ("**BotValve**"). The mixture inside the separator leaves in vapour state through the upper outlet, flowing across the upper valve ("**TopValve**"). In the present flowsheet, the valve stem position is the manipulated variable of the system's controller ("**PI_Pressure**"). A proportional-integral type of control was selected for the present controller model, adding an integral term to the steady-state proportional error. A maximum and a minimum value for both manipulated and controlled variables were specified, with values as shown in table 2:

Table 2- "PI_Pressure" controller specifications

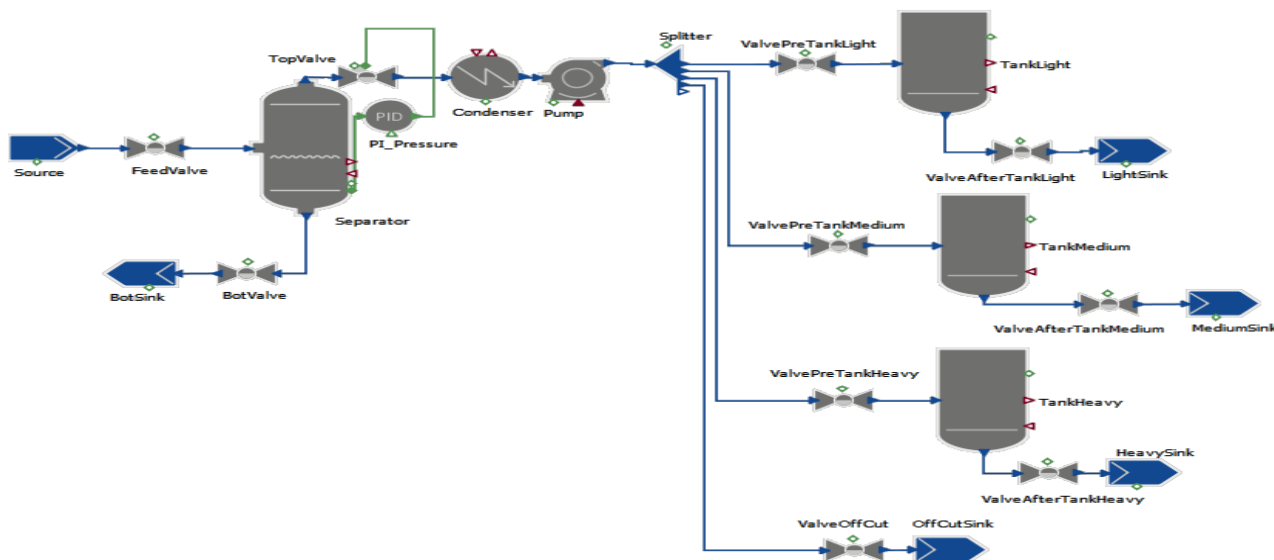| Controller parameters		Value
Controlled variable: "Separator" pressure	Min. value (bar)	0.2
	Max. value (bar)	1
Manipulated variable: "TopValve" stem position	Min. value	0.01
	Max. value	0.99
Controller action:		direct
Proportional gain		144
Integral time constant (s)		10

Figure 1- Separator case study assemble flowsheet

The vapour exiting the separator is promptly condensed in the condenser model ("**Condenser**"). After condensing and subcooling, the distillate is pumped to its final destination, through the pump model ("**Pump**"). All the tank models presented in the current flowsheet have the same dimensions, same initial molar holdup, same pressure and same behaviour: the valve that controls the tank outlet (and named after the respective tank) is closed at all times, allowing a permanent component accumulation inside each tank, depending on which inlet valve is open.

On the current process, two different operating ways were taken into consideration. For the first way, a constant pressure inside the separator was set as the objective of "**PI_Pressure**" controller model, with a setpoint of 0.69 bar.

The second operating way consists of maintaining the temperature constant / under a maximum value, reducing the pressure of the system. The flowsheet for this operating way remains the same, apart from the controller model, which is now manipulating the "**Pump**" energy rate (kJ/s) in order to control the separator's temperature.

The separator model starts at 44% of its total volume. "**FeedValve**" stem position changes its value from 0 (closed) to 0.5 (half-open), allowing the separator to refill its content when the holdup liquid level reaches 1%. The valve stem position closes right after the holdup liquid level inside the separator achieves 95% of the total height. The final destination of the distillate is dependent on its mass fraction. If the distillate mass fraction is 0.45 of cyclohexane, "**ValvePreTankLight**" stem position is switched to 0.5, while the other three valve stem position are changed to zero ("**ValvePreTankMedium**", "**ValvePreTankHeavy**" and "**ValveOffCut**"). The same action is scheduled for a mass fraction of 0.45 for heptane, and 0.40 for Toluene. In the case that two requirements are met, such as 0.45 mass fraction of heptane and 0.40 of toluene, the lightest compound valve has priority.

3.2 – Case study results

The liquid level inside the separator is a non-constant variable during the simulation. Although the liquid level threshold for refilling the separator is the same, the synchronization of both examples is eventually lost (see figure 2).
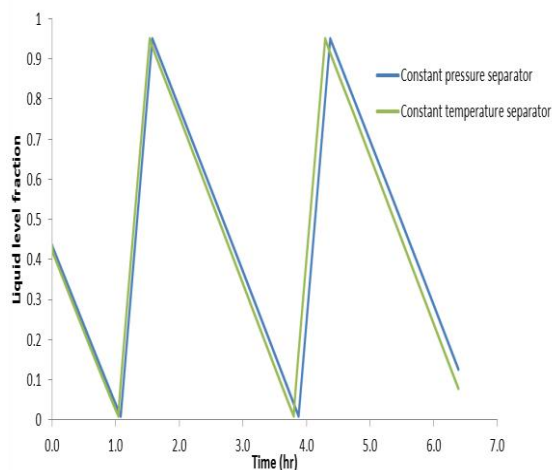

Figure 2- Separator liquid level profile from both simulations

The first case (constant pressure) has an average temperature of 355.5 K and 0.69 bar average pressure. In comparison with the second case (constant temperature), with 352 K of average temperature and 0.615 bar average pressure, the constant pressure simulation will have a slower output of vapour, due to the operating conditions. As it can be seen in figure 3 and 4, there are abrupt changes in the system behaviour twice: approximately at time 1 and 4. During the first re-fill, the "**Source**" feeds the system through the "**FeedValve**", and the temperature, pressure and composition inside the "**Separator**" tend to the source model values. The inlet flow, which influences the re-fill time, is dependent on the pressure difference between the separator model and the source mode. On the other hand, this pressure difference is calculated with the flow coefficient specified in "**FeedValve**"

The dynamic behaviour of the tank models is a direct consequence of the vapour composition and the vapour flow coming from the "**Separator**". All the condensed vapour is accumulated in either a tank model or in the "**OffCutSink**", when the mass fraction requirements are not met. Figure 5 shows the evolution of the mass holdup of all the tank models and the off-cut sink model
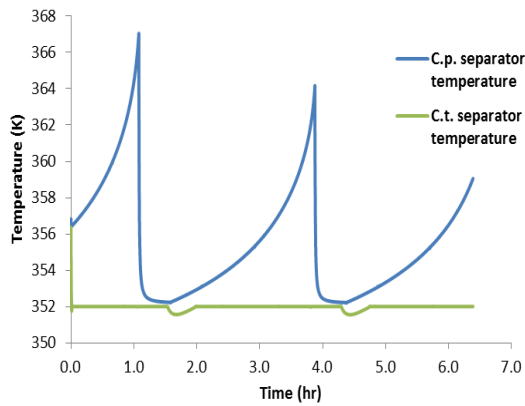
Figure 3- Separator temperature in constant pressure and constant temperature simulations
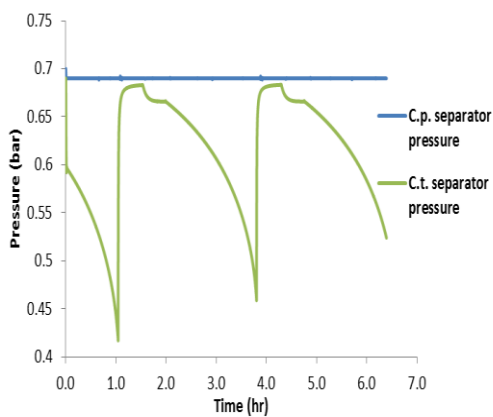

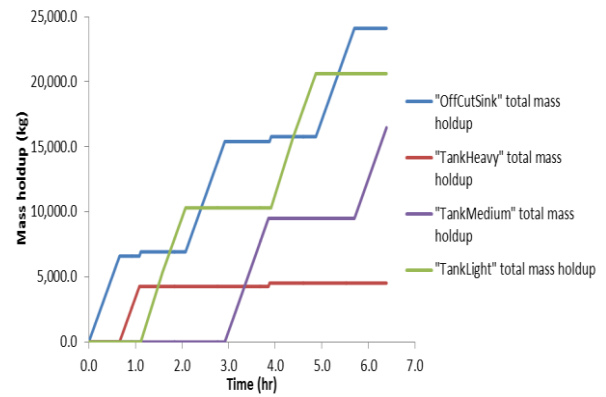*Figure 4 - Separator pressure profile in constant pressure and constant temperature simulations*

Figure 5- Mass holdup in the tank models and off-cut sink in constant pressure simulation

From figure 6 is possible to notice 3 main stem position values, with the values 0.69, 0.56 and 0.63, approximately. The first value is obtained when the "**OffCutSink**" is being filled. This model has a slightly bigger value for the assigned pressure than the tanks (0.8 bar compared with 0.79). Therefore, the stem position changes accordingly to reduce the pressure drop, maintaining the pressure inside the separator constant. This can be observed up to 0.8 hr in figure 28. As soon as the distillate destination changes to any of the other tanks, the value of the stem position acquires values near 0.56, as can be observed 0.8 and 1.1 hr of the simulation. This value has an abrupt change at the end of this interval, consequence to a quick change in the distillate destination, between the "**HeavyTank**", the "**OffCutSink**" and the "**LightTank**". Additionally, the "**FeedValve**" is open to refill the tank during this period of time. While the valve remains open, the manipulated variable value stabilizes around 0.63. The new mixture is more volatile than the previous holdup, therefore requiring a slight change in the "**TopValve**" stem position.
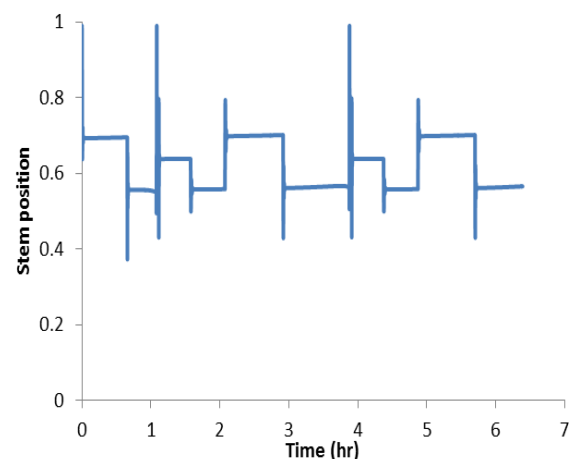
The separation starts with the sink valve model open, as the outlet vapour mass fraction is not rich enough for the "**ValvePreTankHeavy**" stem position to change. That vapour mass fraction, 0.40, is only achieved at 0.7 hr. From that time instant until 1.1 hr, the "**TankHeavy**" is being filled with the separator outlet distillate. As soon as the conditions for the "**FeedValve**" to open are met, a cycle begins:
o In the short time that the separator is being refilled with a new content, the endpoint of the distillate changes twice: first, it is sent to the "**OffCutSink**" when the toluene mass fraction is no longer above 0.4; secondly, when the cyclohexane composition is now the most abundant, the distillate is sent to the "**TankLight**"
o **TankLight**" is filled for about an hour, until the mass composition is no longer 0.45 of cyclohexane. At this point, neither heptane mass fraction is above the mass fraction specification, which means the distillate is carried to the "**OffCutSink**" again.
o This off-cut between the cyclohexane and the heptane lasts for around 50 minutes. At 2.9 hr, the "**ValvePreTankMedium**" is open, and the medium tank is filled
o The transition between the medium component and the heavier one (heptane and toluene) is direct, without off-cut

Figure 6 - Dynamic behaviour of the "TopValve" stem position

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4. BATCH DISTILLATION

4.1 – Model validation

A validation of a batch distillation model was achieved through the comparison between the data available in Bonsfills and Puigjaner [16] and the results obtained in the assembled flowsheet (see figure 8). The pilot column dimensions and operating conditions are presented in table 3.

Table 3- Pilot column dimensions and operating conditions

Column	Column height (m)	3.75
	Number of trays	15
	Inner diameter (mm)	50
	Outer diameter (mm)	90
	Average operating pressure (mmHg)	760
Reboiler	Volume (L)	6
	Maximum heat duty (W)	1400
	Operating heat duty (W)	681.3
Condenser	Operating mode	Total condenser
	Distillate flow (mol/min)	0.19
	Reflux ratio	4
Mixture	Composition	Equimolar
	Volume (L)	6

The model assembled started the simulation in a steady-state continuous distillation, as the initial holdup could not be specified. As a consequence, the component specification in the source model ("**Source_1**") had to be tested and changed until the molar sum of all the trays, condenser and reboiler achieved a composition as close as possible as the Bonsfills experimental data [16].

The column specifications are presented in table 4. All the valve models (**Valve_1, Valve_2** and **Valve_3**) have been set to operate in mass and energy balance mode with a pressure drop specification of 0.1 kPa, and a stem position of 0.2.

For the current flowsheet, two PI controllers were added in order to maintain the pressure inside the column and the distillate flowrate constant. The first controller (PI_1) controls the second stage pressure (1st column tray) using the condenser heat duty as manipulated variable. Differently from flow-driven mode, the condenser heat duty is a specification from this operating mode, which does not assure total condensing of the inlet vapour in the condenser, if left untouched. Using this variable as PI_1 manipulated variable, both constant pressure and total condensing problems are solved. The second controller (PI_2) controls the distillate flowrate using the Valve_2 stem position as a manipulated variable, with values varying between 0 and 1 (fully closed and fully open, respectively)

For the validation, the simulation begins in steady-state with bottom and top liquid being recovered. At time 0

Table 3 –Column 1 specifications

Column	Column diameter (cm)	5
	Plate efficiency (%)	80
	Plate spacing (cm)	25
	Stages	17
Condenser	Diameter (cm)	8
	Volume (L)	1
	Liquid level (cm) / liquid fraction	5.97 / 0.30
	Inlet valve flow coefficient (kg s^{-1} Pa^{-1})	1 x 10^{-4}
	Stem position of the inlet valve	0.5
	Stem position of the reflux valve	0.5
	Reflux ratio normalized	0.8
	Operating mode	Total condenser
Reboiler	Diameter (cm)	25
	Volume (L)	0.19
	Liquid level (cm) / liquid fraction	12.12 / 0.85
	Boilup ratio normalized	0.8
Trays	Active area fraction	0.8
	Hole area fraction	0.1
	Weir fraction	0.7
	Weir height (mm)	9.55
	Tray thickness (mm)	2
	Hole diameter (mm)	4.5
Pressure	Dry vapour press. drop correlation	Bernoulli
	Aerated liquid press. drop correlation	Bennett
	Clear liquid height correlation	Bennett
	Initial guess for press. drop per stage (bar)	4 x 10^{-4}

(after switching to flow-driven) Valve_1 and Valve_3 stem position (figure 8)were set to the value of zero. The same value has been used for PI_2 setpoint, closing the valve in less than 4 seconds. After 100 seconds of simulation, the reboiler heat duty value is changed to match the same value as Bonsfills' heat duty (681.3 Watts). The simulation runs for another 1500 seconds in order to stabilize. At time 1600, PI_2 setpoint is changed to match Bonsfills' distillate flowrate (0.19 mol/min, 0.0114 kmol/hr) and the simulation runs until there is no holdup inside the reboiler. Distillate molar faction and trays temperature profiles were then compared.
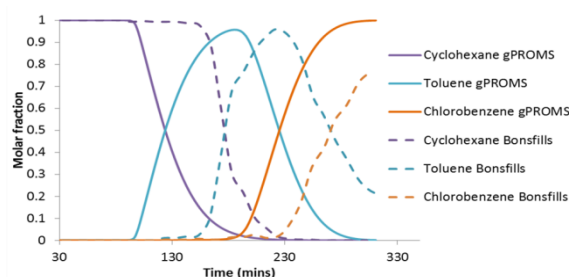

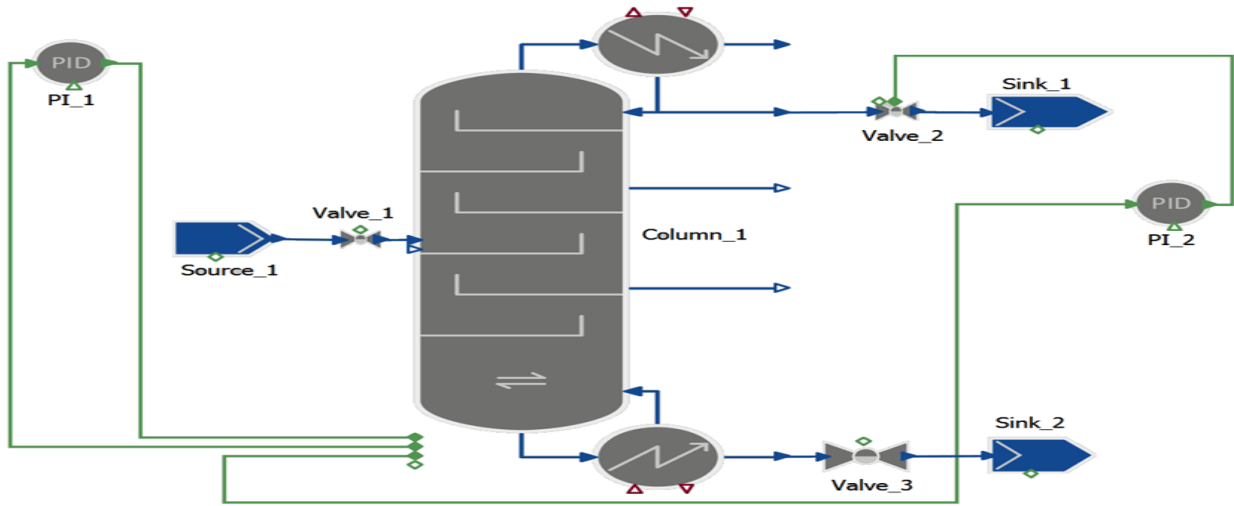*Figure 5 - First simulated distillate molar fraction profiles*

Figure 6 - Distillation model assembled flowsheet

Figure 7 shows a position shift for the profiles, between Bonsfills' results and the simulated ones [16]. The shape between the curves is pretty similar, indicating a good understanding and good results for the component interactions. However, the same figure appears to indicate that a different component composition has been loaded in the same column. The integration of Bonsfills operating at a constant distillate rate of 0.19 mol/min is presented in table 5:

Table 5 – Integration from Bonsfills distillation profiles

	Molar holdup (mol)
Cyclohexane	27.92
Toluene	17.90
Chlorobenzene	7.82
Total	53.84
Difference between initial holdup and distilled mols	3.09

Table 5 clearly shows that there is no equimolarity in the distillation profile curve presented in [16]. Therefore, a different simulation has been prepared. This simulation, named SIM-2, has the same initial volume, but the composition is the same as table 5. The missing moles were arbitrarily distributed between the heaviest and the intermedium component (80% and 20%, respectively).

The results, shown in figure 9 show a good prediction of the mixture's behaviour. The cyclohexane curve has the best behaviour prediction, slightly missing the time period when the toluene composition gets richer, in the distillate. The interactions between cyclohexane and toluene are well projected by the properties package Multiflash.
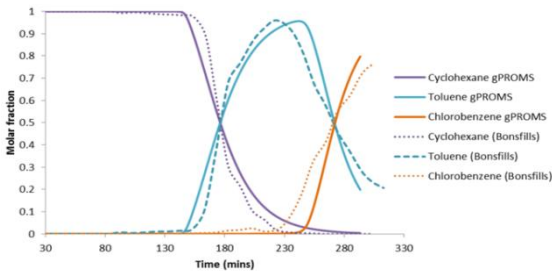

Figure 7- Simulated distillate molar fraction profiles for SIM-2

The maximum purity achieved for toluene is the same in both cases (Bonsfills results and gPROMS simulation), but a time shift of 10 minutes can be observed between the 0.96 molar fraction maximum value. As it is observable, the maximum

purity achieved by gPROMS for the chlorobenzene component is far greater than the results shown by Bonsfills. The deviations verified are mostly under 0.05. In fact, the average deviation value for cyclohexane, toluene and chlorobenzene is 0.03, 0.05 and 0.03, (respectively), and the maximum deviation value verified for each of the three components is 0.16, 0.22 and 0.24.

4.2 – Sensitivity analysis

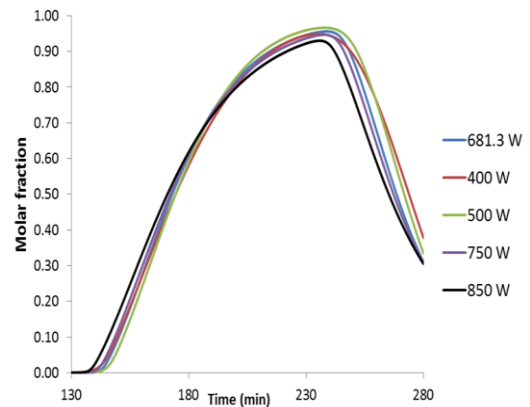The variables reboiler heat duty, the reflux valve stem position

Figure 8 - Toluene molar fractions for different heat duty inputs

and the distillate flow setpoint from PI_2 controller were changed to understand their impact in the simulation results.

The reboiler heat duty has been changed for the values 400, 500, 750 and 850 W, and its influence in the distillate composition studied. Figure 10 shows the toluene composition profile behaviour for different heat duty inputs. It is possible to notice the difference between the maximum purity achieved for the different heat duty input values. Remarkably, the highest purity does not belong to the lowest heat duty value, 400 W, but to the second lowest, 500 W.

Taking a closer look at the condenser liquid holdup fraction profile in figure 11, it can be noticed that for higher values of heat duty there is an increment in the reboiler liquid level after the column stabilization. An increment in the reboiler heat duty will increase the vapour flow leaving the reboiler. This vapour flow increment is responsible for a temporary raise in the system pressure. Controller PI_1 manipulated variable will change accordingly, decreasing the condenser heat duty. During this short period of time, the increment in the system pressure will also decrease the molar flowrate returning to the column from the condenser. Additionally, the decrease of the condenser heat duty will be responsible for a subtle rise in the molar flowrate entering the condenser. As a consequence, the liquid level of the condenser increases until the liquid's hydrostatic pressure is enough to stabilize the whole column.

The distillate flowrate setpoint has been changed to 0.006, 0.009, 0.02 and 0.03 kmol/h values. The main difference between the different distillate flowrates is the distillation time required for the whole simulation. A bigger flowrate will empty the column faster, and therefore reduce the distillation time, as can be observed in figure 12.
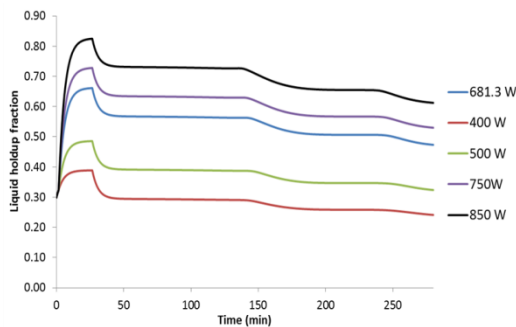
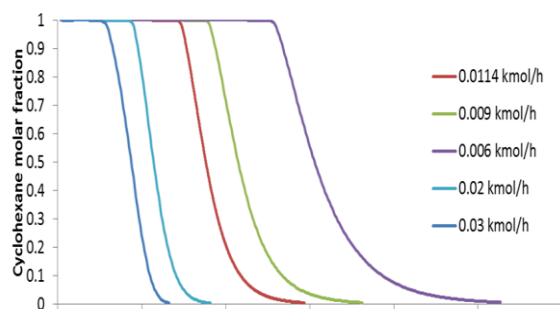*Figure 9- Condenser liquid holdup fraction for different heat duty inputs*

Figure 10 - Cyclohexane molar fraction profiles for different PI_2 controller setpoints

The changes in the distillate flowrate can extent the distillation from 293 minutes to 527, or reduce it up to 133 minutes (30 minutes of stabilization included). In terms of system optimisation, the distillate flowrate is one of the most important factors in time mitigation and/or time control. All of the PI_2 controller setpoints simulations were realized with a fixed value for the reflux valve stem position, and therefore the reflux ratio profiles are different for each setpoint value. The average normalized reflux ratio for each different distillate flowrate setpoint is presented in table 6.

Table 6 – Average normalized reflux ratio for each different PI_2 controller setpoint

PI_2 setpoint (kmol/hr)	Average normalized reflux ratio
6.00×10^{-3}	0.92
9.00×10^{-3}	0.88
1.14×10^{-2}	0.85
2.00×10^{-2}	0.74
3.00×10^{-2}	0.61

The reflux valve stem position (RVSP) has been changed to 0.35, 0.425, 0.575 and 0.65, and the distillate molar fraction profiles analysed. Different values for the RVSP will have an impact in the initial condenser holdup value. This will be reflected in the distillate molar fraction profiles, as can be observed in figure 13:
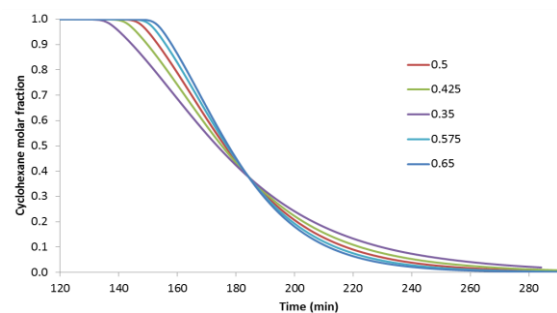
Figure 11- Cyclohexane fraction profile for different reflux valve stem positions

An additional sensitivity analysis has been performed. The reboiler heat duty and the RVSP have been changed in a step behaviour, in order to analyse the system behaviour and possible mathematical integration problems, in the distillation model. Figure 14 shows the influence of the reboiler heat duty step variations:
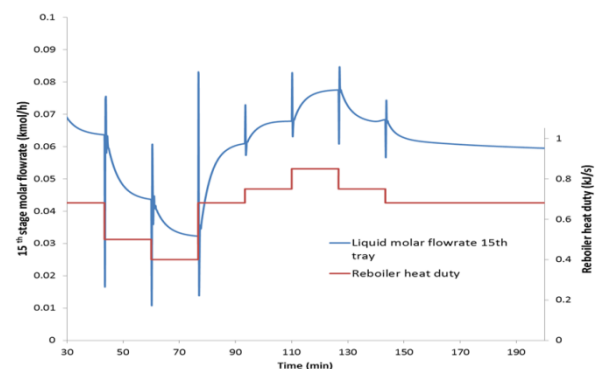
Figure 12 - 15th tray liquid molar flowrate for reboiler heat duty step variations

As can be observed, a spike arises each time the reboiler heat duty input changes. Such sudden variations carry difficult integration problems for the gPROMS® ProcessBuilder simulation solver. In order to solve and overcome the problems presented in figure 14, a new controller model was added to the flowsheet, as well as an Energy Source model and an Energy Sink model. Figure 15 presents the system behaviour with the implemented modifications:
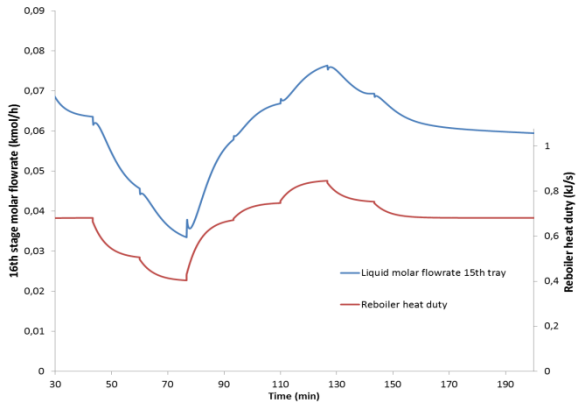
Figure 13- 15th tray liquid flowrate profile for reboiler heat duty step variations, with the implemented modifications

4.3 – Optimisation

An optimisation problem applied to the SIM-2 system, with the changes presented in sub-chapter 5.2.5, is solved in a constant-piecewise operation. The Capacity factor (CAP) [14] presented by Luyben has been set as base for optimisation objective. Three different optimisations were realised: optimisation of the lightest component distillation, optimisation of the lightest and medium component distillation, and optimisation of the whole distillation.

The objective function is hereby presented:

$$Max_{mol} = \frac{Product_{recovery}}{Time} \qquad \text{(eq. 2)}$$

where the *Product*_*recovery* is the molar holdup recovered from the initial column holdup within purity specifications (mol), and *Time* is the simulation time (hours). The *Product*_*recovery* variable is different for each of the three optimisations, depending on which component recoveries are being maximised.

The recovery of each component is calculated by adding the following equation:

$$Light_{acum}('n') = \int Distillate_{flowrate} * Distillate_{Purity}('n') \\ * Constraints('m') \, .dt$$

(eq. 3)

where *Disitillate*_*flowrate* is the distillate molar flowrate leaving the condenser (mol/hr) and the *Distillate*_*purity*('n') is the molar fraction of the *n* component. *'m'* stands for the recovery period, either light component recovery period or medium component recovery period. For the full distillation problem, the chlorobenzene recovery happens in the reboiler. The variable *Constraints('m')* acquires values between 0 and1, and has the objective to nullify or validate the *Light*_*acum* ('n'). That objective is achieved by using a modified hyperbolic tangent equation in which "true / false" statements are converted into 0 or 1 numerical values:

$$Constraints('m') = \frac{\tanh[\,(Ver_{DistPurity}('m') + Ver_{TankPurity}('m') - 0.5) * \beta]}{2} + 0.5$$

(eq.4)

where *Ver*_*DistPurity* ('m') and *Ver*_*TankPurity* ('m') are verification variables regarding the distillate purity and the accumulation purity, depending on which component is being distilled. These variables have values between 1 and 0. β is an adimensional tuning factor responsible for a faster function change. This tuning factor was set as 500.

For the present system, the decision variables optimised were the same as in sub-chapter 3- sensitivity analysis: PI_3 setpoint (reboiler heat duty, kJ/s), PI_2 setpoint (distillate valve stem position) and reflux valve stem position. These variables are fixed at the beginning of the simulation, replicating the same behaviour as previous chapters. The decision variables optimisation is realised after the distillation valve opening, at 1601 simulated seconds. Table 7 presents the optimisation constraints for the different simulation problems:

Table 7 – Optimisation constraints for the different simulations

	Minimum recovery (mol)		Minimum purity	
Cyclohexane	24		0.98	
Toluene	8		0.93	
Chlorobenzene	4		0.91	
Light component optimisation				
Min. interval duration (s)	Max. interval duration (s)	Min. total time (s)	Max. total time (s)	
300	3000	3000	16000	
Medium and light component optimisation				
Min. interval duration (s)	Max. interval duration (s)	Min. total time (s)	Max. total time (s)	
300	4000	4000	17000	
Full distillation problem				
Min. interval duration (s)	Max. interval duration (s)	Min. total time (s)	Max. total time (s)	
300	5000	6000	17000	

For the light component distillation problem, the objective function improved from an initial value of 9.50 mol/h to 19.92 mol/h. The recovery of the light component, cyclohexane, had a slight increase in its value. However, the distillation time reduction has been huge, as can be observed in figure 16:
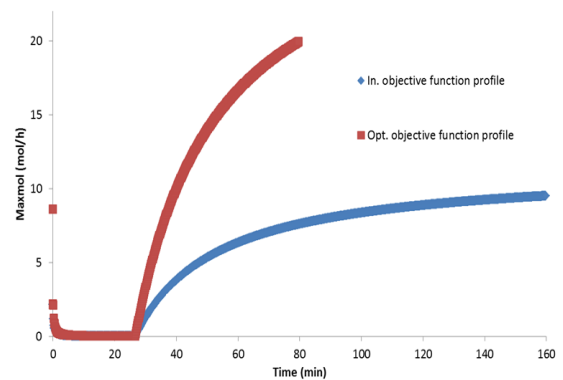

Figure 14- Initial objective function profile and optimised objective function profile for light component distillation problem

The MaxMol variable stabilizes after the fourth control interval. In fact, the reflux valve stem position requires three time intervals until it achieves its maximum position, and stabilizes. The reboiler heat duty has an abrupt change at the last time interval, and it remains in the lowest possible value until a hypothetical off-cut, as in Medium and Light component optimisation. Therefore, 4 is the minimum number of required control intervals to achieve the optimal solution for the Light component optimisation, as can be noticed in figure 17:
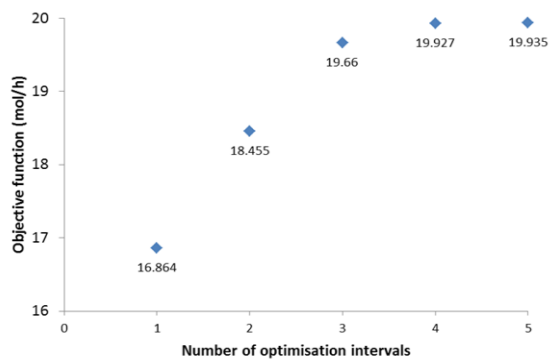
8

Figure 15 - Optimised objective function values for different control time intervals, for light component recovery problem.

For the medium and light component distillation problem, the recovery of both components has increased. Toluene, which is considered to be the most difficult component to separate in this mixture, has more than double the amount of recovered product. Also, the amount of wasted product (off-cut) has hugely decreased, while the distillation time has decreased, as it is shown in figure 18.

For the full distillation problem, the objective function profile is observable in figure 19. The two initial maximum values are the initial recovery of cyclohexane, followed by a temporary purification of the system, and the distillation of the remaining in-spec cyclohexane.
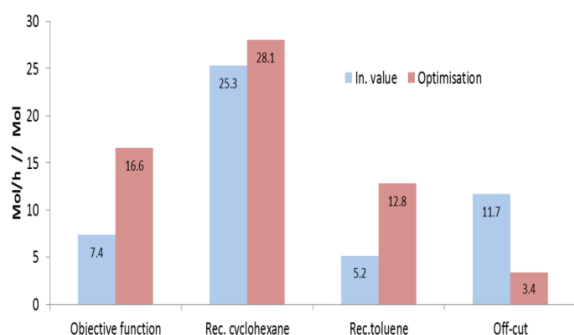

Figure 18-Comparison between initial and optimised values for toluene and cyclohexane recovery, off-cut, and the objective function

However, the optimisation has matched this time event with the enrichment of chlorobenzene in the reboiler. By the time the recovery of toluene is finished, the chlorobenzene purity has reached the desired 91%. Comparing with the initial MaxMol profile, the toluene distillation was still ongoing when the chlorobenzene purity achieved 91%.
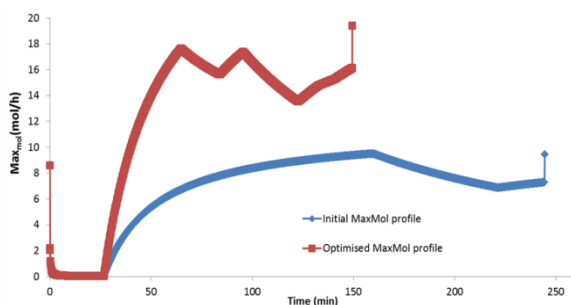

Figure 19- Initial and optimised objective function profile for full distillation problem

It is now possible to compare the distribution of the initial holdup through the different components recovery and the off-specification product. Figure 20 compares the distribution of the initial holdup at the end of the distillation for the initial simulation and figure 21 compares the distribution of the initial holdup at the end of the distillation for the full optimised distillation:
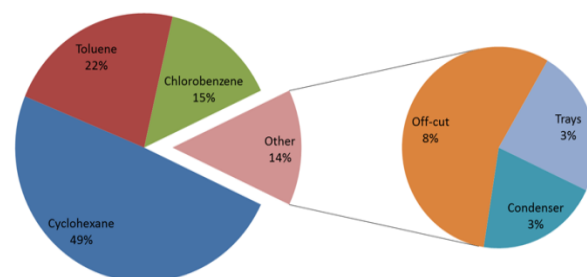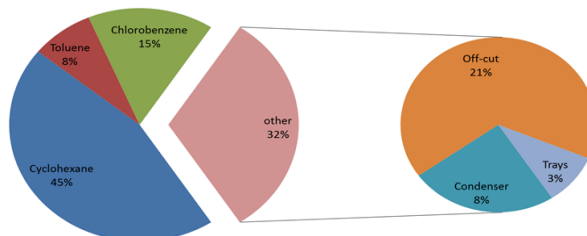

Figure 21 - Final distribution of the initial holdup, in the full distillation optimisation

5. CONCLUSIONS AND FUTURE WORK

The aim of this work was to simulate and validate the batch distillation model developed in this work, and further including proper optimisation problems and solutions.

Using the case study of the present work as an example: one separator has been assembled to separate a ternary mixture at either constant pressure or constant temperature. All the other initial variables and conditions are the same. The most impressive results are the intense changes in the vapour outlet flow from both separators. In terms of separation, the results would be the same as if this separation occurred in a one-stage distillation column. For the specification arbitrarily chosen, the off-cut sink holds 37% of the entire product that was separated. The light component, cyclohexane, only achieves 31%, followed up by the 25 % of heptane and 7% of toluene. However, the initial holdup is different from the feed specification. Thus, a steady cyclic state is not achieved in this study case.

For a complete model validation, simulation results have been compared with experimental data available in the literature. From the results obtained, the operating strategy chosen for the modelled system is different from the operating policy in the available data. Clearly Bonsfills operated the pilo column with a constant reflux ratio instead of a constant distillate flowrate. However, after adapting the system to the new operating strategy, the simulated composition profiles were very similar to the experimental values.

The sensitivity analysis realized in this work has revealed that changes in the heat duty and in the reflux valve have a huge impact in the distillate purity. Additionally, it was possible to

detect and correct mathematical problems within the distillation model thanks to a step sensitivity analysis realized in two of the three analysed variables.

A set of optimisation problems have been addressed: optimising the recovery rate of the light component, optimising the recovery rate of the medium and the lightest component, and optimising the recovery rate of the whole distillation. These problems shared the same purity constraints for the respective components, and the initialisation procedure was equal for all the three problems.

The optimal operating policy for the system has been found, considering the heat duty input, the reflux valve and the distillate flowrate as control variables. The minimum number of control intervals has also been found for the first two problems, being 5 and 8, respectively. Using the capacity factor (CAP) as an objective function, the results were 19.9, 16.6 and 19.4 mol/hr respectively. In all the optimisation problems referred, the time and energy consumption have been reduced.

Regarding the future perspectives of this work, there are three important points that should be revised:

o Regarding the system assembly, column models require special attention in the initial system definition. A initial holdup specification might be considered, with an assumption of a mixture distribution between the column stages;

o For the system assembled, further work can be done in the optimisation of the distillation. Not only by changing the objective function to a profit one, but also to analyse the recovery of the of-cut and tray and condenser holdup;

o The comparison of a new operating strategy for the assemble model can be of great interest in terms of additional validation. As it was stated before, the operating policy followed in this thesis is different than the one adopted in the experimental results.

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