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Research note

Comparing and controlling of three batch distillation column configurations for separating tertiary zeotropic mixtures

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Batch distillation;
PI controller;
Dynamic simulation;
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Abstract This paper concerns the simulation and control of multi-vessel batch distillation configurations for separating tertiary zeotropic mixtures. Three configurations, namely, conventional middle vessel, modified middle vessel and rectifier column were selected. Unlike the previous works, a detailed model without assumptions of constant level, pressure and boilup rate was used. This model was simulated in Aspen Hysys 2009 software. In addition, a control structure which has one pressure and one/two level controllers was selected for each configuration. The results indicate that the selected control structure has an acceptable performance and the modified middle vessel configuration needs the least batch time and energy consumption. Finally, the performances of level control and temperature control strategies were compared. The results show that the level control strategy has better dynamic performance and needs lower batch time, but with uncertain feed composition, the temperature control structure is preferred.

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1. Introduction

Batch distillation is the oldest separation process and the most widely used unit operation in the batch industry. Batch distillation is used more in comparison with continuous distillation when high-value-added, low-volume chemicals must be separated, so it is mainly used in specialty chemical, biochemical and pharmaceutical industries. Although this kind of separation has lower energy efficiency than continuous distillation, it has been widely considered because it is more flexible, simple and needs lower capital cost.

A multi-vessel batch distillation column which is a combination of a batch rectifier and stripper columns can be used to separate a ternary mixture. A middle vessel column was mentioned by Robinson and Gilliland [1] and was first analyzed for

purifying binary mixtures by Bortolini and Guarise [2]. For the first time, Hasebe et al. [3] charged a ternary mixture to the middle vessel, so the light and heavy components accumulated at the top and bottom of the column, respectively and he stopped the operation when the intermediate component reached its desired purity in the middle vessel.

Moreover, a cyclic two-vessel column which is only a rectifier column may be used to separate a mixture. For example, for a ternary mixture, the component with the lowest boiling temperature will be removed from the top vessel of the first cycle and the other components will be separated in the second cycle. We know that for splitting a mixture with N species to its constituents simultaneously, $N - 1$ columns must be used. Hasebe et al. [4–6] compared multi-vessel column with rectifier column and found out that energy consumption in multi-vessel column is about half of the cyclic two-vessel column. Hilmen [7] enforced that multi-vessel column needs less time than two-vessel column. Therefore cyclic method is mostly appropriate for small laboratories.

Two basic modes of batch distillation are: (1) constant reflux and (2) variable reflux which result in variable distillate composition and constant distillate composition, respectively. The third operating model of a batch distillation; optimal reflux or optimal control, is neither constant nor variable reflux, but it is between the two mentioned methods.

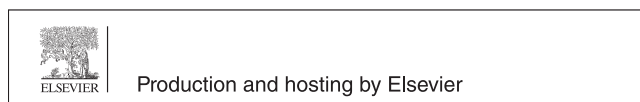
Similar operating modes are also observed in the emerging batch distillation columns. For example, a stripper can also

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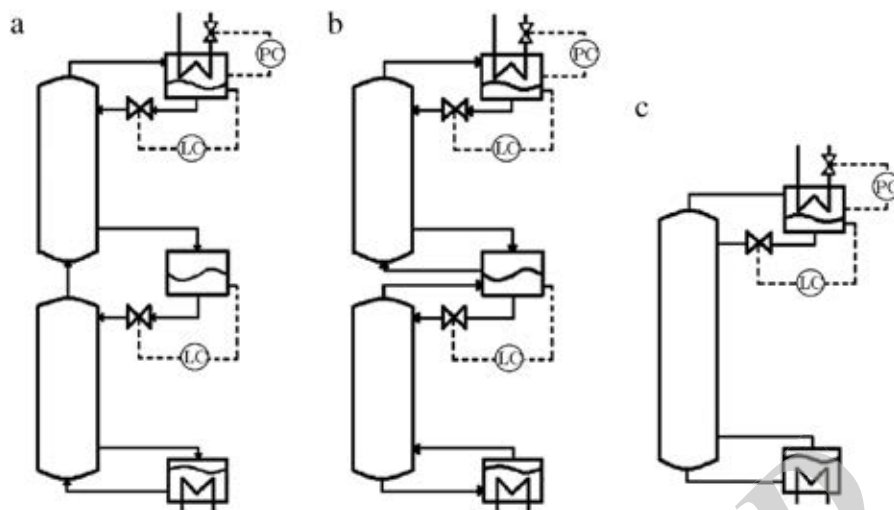


Figure 1: (a) Conventional middle vessel column; (b) modified middle vessel column; and (c) rectifier column.

have three operating modes: constant reboil ratio, variable reboil ratio and optimal reboil ratio. For a middle vessel column, the combination of the three refluxes and three reboil modes results in nine possible operating policies. The operating modes of a multi-vessel column can be derived based on the middle vessel column, but this column configuration requires additional considerations with respect to the operating variables, such as the holdup in each vessel. The total reflux mode can be also considered especially in the middle vessel and multi-vessel columns. Noda et al. [8] showed that when total reflux mode with optimal conditions is applied in a rectifier column, it has less energy consumption.

Three configurations for separating a ternary mixture are shown in Figure 1. In the conventional type, the middle vessel only receives the liquid flow from the rectifying part and the vapor from stripping section directly goes to the bottom of the upper column. But in the modified case, the vapor stream first enters into the middle vessel and then goes to the upper column; hence the middle vessel behaves like an equilibrium stage.

Two different strategies are selected to “control” the total reflux multi-vessel batch distillation column. Hasebe et al. [4] calculated the final holdup in each vessel and then used a level control system to keep the holdup in each vessel constant. His control structure involved the optimization of the vessel holdups based on composition measurement. In other papers, most authors like Skogestad et al. [9] measured a stage temperature for each section of the column and designed a feedback temperature control structure wherein the reflux flows were as the manipulated variables. Also Wittgens and Skogestad [10] compared their experimental works with simulation results. They assumed that the mixture temperature on each stage is the molar average of the pure components boiling temperature; they used this assumption to specify the set point for each controller.

In this paper the energy consumption and operation time of conventional, modified and rectifier batch distillation columns are compared based on a level (and pressure) control strategy. Then the controllability and performance of the level control strategy is compared with the temperature control strategy, especially when the feed has uncertain composition.

2. Simulation

2.1. Thermodynamic model validation

Equilibrium data are very important for simulation, design and optimization of separation operations. In fact, the results of a simulation can be true when the thermodynamic model estimates the behavior of the mixture correctly.

Gruetzmann and Fieg [11] used NRTL model for the ternary mixture of hexanol, octanol and decanol and showed that the model results and experimental data are nearly the same. Based on their study, the same components and thermodynamic model are applied. In Aspen Hysys, the heat of mixing is ignored when the activity models are used. Although it is not an important problem for the studied mixture in this paper (nearly the same chemical species), the components and thermodynamic model (NRTL) were added from Aspen Properties 2009 which can estimate the heat of mixing.

2.2. Simulation details

Some simplifier assumptions, such as constant relative volatilities, constant pressure and vapor flow, in the columns were used by previous researchers [11]. However these assumptions may cause some deviation from real conditions. So we tried to simulate the actual behavior of the operation by using Aspen Hysys 2009 software and not applying these assumptions.

For purifying the alcoholic mixture of hexanol, octanol and decanol, three configurations including conventional middle vessel, modified middle vessel and cyclic rectifier column are simulated (Figure 1). Different variables like operation conditions, number of stages and etc. have significant effects in the results. These details that are related to optimization problems were considered by Furlonge et al. [12], Low and Sorensen [13–15].

Theoretically the number of stages per section affect the batch time. The batch time and consequently, the operating costs decrease by increasing the number of stages but on the other hand, the initial investment costs grow. So, in our simulation after considering the effect of equilibrium stages on the separation, sufficient equilibrium stages are chosen roughly

Table 1: Columns specifications.

Column sequence	Conventional	Modified	Rectifier	
			Cycle 1	Cycle 2
Type	Packed	Packed	Packed	Packed
Vessel volume (m ³)	1.618	1.618	2.427	1.715
Equivalent number of stages ^a	$N_r = 11$ $N_s = 11$	$N_r = 11$ $N_s = 10$	$N = 22$	$N = 22$
Column diameter (m)	0.6	0.6	0.6	0.6

^a N_r and N_s are the number of stages in rectifying and stripping sections, respectively.

Table 2: Physical properties.

Property	C ₆ H ₁₄ O	C ₈ H ₁₈ O	C ₁₀ H ₂₂ O
Boiling temperature at 103 kPa (°C)	157.5	196.1	231.7
Molecular weight	102.2	130.2	158.3
Molar density (kg mol/m ³)	6.724	5.139	4.107

to achieve the minimum time required and simultaneously prevent using additional stages which can only cause to increase the initial investment costs. Thus the total number of trays for conventional method is 22. Whereas the middle vessel in the modified sequence behaves like an equilibrium stage, the number of trays in stripping section is one less than the conventional but the rectifying sections are similar. Also for comparison, equal stages for middle vessel column and cyclic two-vessel column are applied. Table 1 shows the specifications of three configurations.

Initial feed distribution is another operative parameter. Hasebe et al. [4–6] and Furlonge et al. [12] have proved that in many cases when the initial feed is charged to the reboiler vessel, the operation will be close to the optimum condition. Based on simulation experiences, the worst status is when it is fed to the middle vessel whereas equivalent distribution in the vessels is close to “initial feed in the reboiler” and controlling the process will be easier. For cases that the components are sensitive to the heat, it is not proper to apply “initial feed in the reboiler”. In all simulations, equal mass of liquid was charged into the three vessels of the middle vessels configurations, but 30% and 70% liquids were charged into the top and bottom vessels of the first cycle of cyclic column, respectively. The physical properties of the components that are used to specify the liquid volume accumulated in the vessels are presented in Table 2.

At the start of the process, it is assumed that the column has atmospheric pressure. And the trays and vessels have the boiling point temperature of the initial feed mixture. The initial values of three configurations are summarized in Table 3.

2.3. Control structure

Controlling the temperature of a tray in each section of the column by adjusting the reflux flow is the most general

method as the control strategy. To simulate the batch operation and to test the temperature control structure, most of the researches used a simple model and assumed that the pressure is constant. To approach the real process behavior, unlike the other researchers, a complete model for controlling the system is used. After considering different control strategies, a control structure which is more appropriate for the real model is selected. In this structure, the column pressure is controlled by adjusting the condenser duty. In addition, the reflux flows are used to control the top and middle vessel levels (level control strategy).

Figure 2 shows the simulated flowsheets of the considered configurations and their control structures. The type of all controllers is PI. Different methods like Ziegler–Nichols may be used to determine the parameters of the controllers, but it is not a good manner because the process must stand at its instability limit. To prevent it, Haggland and Astrom [16] presented the Relay-Feedback test in 1984. They have put a relay instead of the controller in the control loop; the relay has special amplitude and its output can only change from the positive to the negative amplitude value based on the output of the control loop. This act is repeated until the error signal oscillates with a constant period. Finally, the controller gain and time constant can be tuned based on the relay amplitude and the obtained period and amplitude of the error signal. It is clear that the process must be at steady state condition to determine the controller parameters. Because of the dynamic characteristic of batch distillation, initially the controllers are tuned by trial and error. Then at the end of the process when it becomes steady, the relay-feedback auto tuning is used and the simulation is restarted. Because of the interaction, the controllers are tuned sequentially. Controller in the faster loop is tuned first, so pressure, top level and middle level controllers are tuned, respectively. The calculated parameters of the controllers are presented in Table 4. To calculate the gain (K) of each controller, the process variable (PV) and output (OP) of controller are normalized between a minimum and maximum value (PV_{\min} , PV_{\max} ; OP_{\min} and OP_{\max}).

3. Results

In all simulations, a feed with equal mass fraction of hexanol, octanol and decanol is used.

Table 3: Initial conditions.

Column sequence	Conventional	Modified	Rectifier	
			Cycle 1	Cycle 2
Feed (kg)	1668.9	1668.9	1668.9	1112.4
Temperature (°C)	176.1	176.1	176.1	176.1
Pressure (kPa)	101.3	101.3	101.3	101.3
Section holdup (m ³)	8.836×10^{-2}	8.836×10^{-2}	8.836×10^{-2}	8.836×10^{-2}
Top vessel holdup (kg)	556.3	556.3	556.3	556.3
Middle vessel holdup (kg)	556.3	556.3	–	–
Bottom vessel holdup (kg)	556.3	556.3	1112.4	556.3

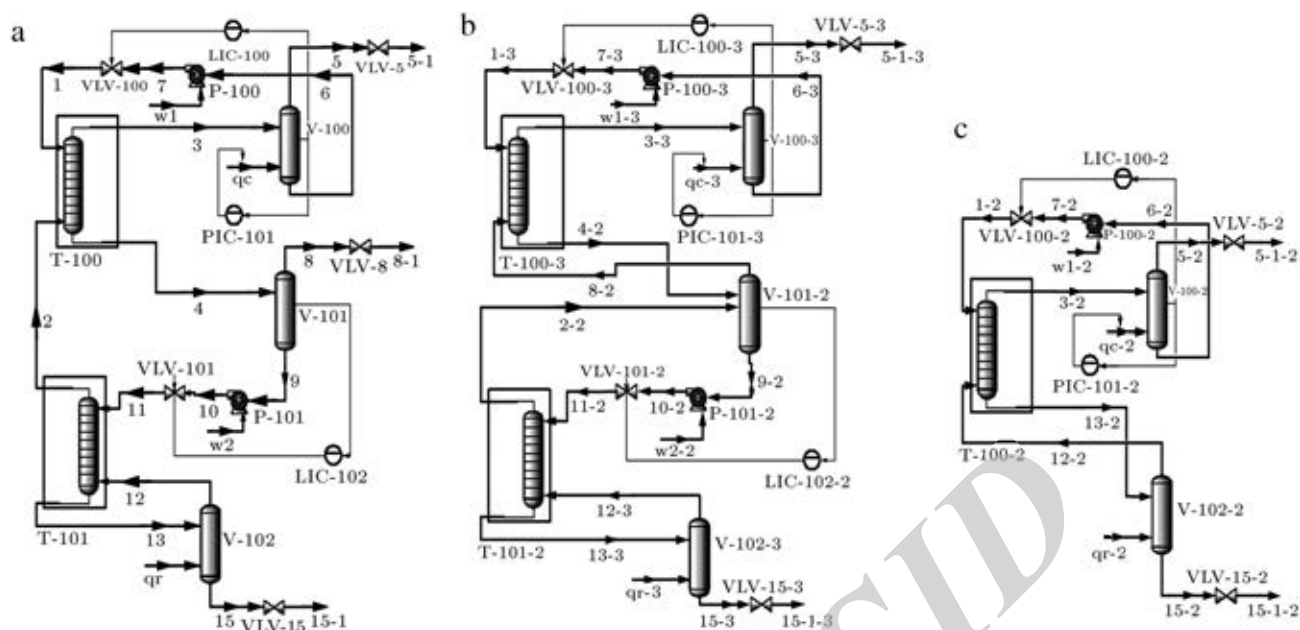


Figure 2: Simulated PFDs in Aspen Hysys 2009. (a) Conventional; (b) modified; and (c) rectifier column.

Table 4: Controllers parameters of the level control strategy.

Controller	Pressure ^{b,c}			Top level ^d			Middle level ^d		
	K	τ_l (min)	SP ^a (kPa)	K	τ_l (min)	SP ^a (%)	K	τ_l (min)	SP ^a (%)
Conventional	4.48	0.678	103	14	1.15	51.88	7.5	2.2	53
Modified	9.28	0.44	103	12	1.26	51.88	4	6	53
Cycle 1	9	0.3	103	25	2.1	35.02	–	–	–
Cycle 2	4.5	12	103	10	6.5	49.57	–	–	–

^a SP: Set point of each controller.

^b PV_{min} and PV_{max} for pressure controller are 80 and 120 kPa, respectively.

^c OP_{min} and OP_{max} for pressure controller are –155.56 and 0 kW, respectively.

^d All other level controllers have OP and PV in the range of 0%–100%.

Table 5: Column sizing factors.

Physical properties					
Liquid			Vapor		
Viscosity (cp)	Density (kg/m ³)	Flow (kg/h)	Viscosity (cp)	Density (kg/m ³)	Flow (kg/h)
0.2114	669.8	834	7.553×10^{-3}	3.432	824
Design parameters					
Packing type	HETP (m)	Foaming factor	Max flooding		
Berl saddles (Ceramic, random) 1 in	0.4572	1	0.6		

3.1. Column diameter and number of stages

An important factor which is used to specify the column diameter is the amount of vapor flow. Based on our results, the maximum vapor flow in the column occurs at the end of the process, although it has large oscillation when the process starts. In the entire configuration, the vapor molar flow is almost the same because of the similar heat duty of the reboiler. For instance, in the modified system the maximum vapor molar flow is about 6.46 kmole/h which is used to calculate the column diameter. Packed columns are chosen for all configurations. The specifications which are necessary to calculate the diameter are summarized in Table 5.

Another variable which has a large role in determination of batch time and operating costs is the number of stages or the height of the packed column. To consider the impact of the mentioned factor, equivalent stages for different heights of the modified packed column are calculated and the results are shown in Figure 3. As it can be seen, the batch time decreases with increasing the column height (number of theoretical trays). An economical study is required to find the optimum number of theoretical trays, but roughly based on Figure 3, 22 theoretical trays are selected for all simulations.

3.2. Composition profiles and energy consumption in three configurations

The composition profiles in the vessels of conventional and modified batch distillation columns are shown in Figures 4 and 5, respectively. In the composition profile of the bottom vessel, the lightest component concentration decreases rapidly and the concentration of the middle component starts to increase at first, then after some time, returns to the expected way. This is because at the start, the reboiler duty causes to raise the vapor rate production which is rich in hexanol and the liquid becomes concentrated of octanol and decanol. For the top vessel, there is no unusual variation, but in the middle vessel, a time delay can be seen in the beginning of the composition changes (see figures 4(b) and 5(b)). The time that lasts to receive

Table 6: Simulation results.

Column	Reboiler duty ($J \times 10^6$)	Condenser duty ($J \times 10^6$)	Time (min)	Final compositions (molar)	
Conventional	2690.7	2595.8	504.5	[0.9985, 0.99, 0.9991]	
Modified (LC) ^a	2152	2058.6	403.5	[0.996, 0.99, 0.9922]	
Rectifier	Cycle 1	1733.3	1659.3	325	[0.99, 0.535, 0.465]
	Cycle 2	1578.7	1550	296	[-, 0.99, 1]
Modified (TC) ^b	2453.3	2357.4	460	[0.991, 0.99, 0.9977]	

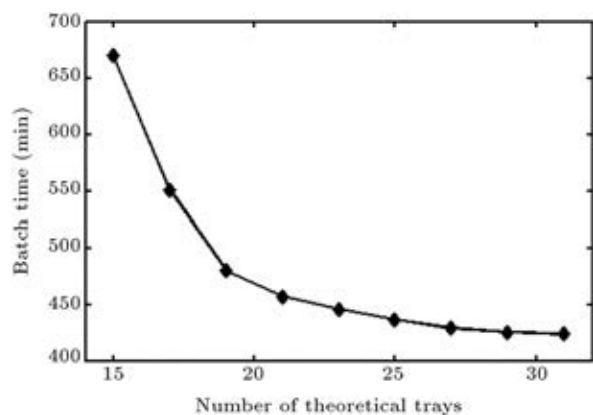
^a Modified with level control structure.^b Modified with temperature control structure.

Figure 3: Impact of number of stages on batch time for modified configuration.

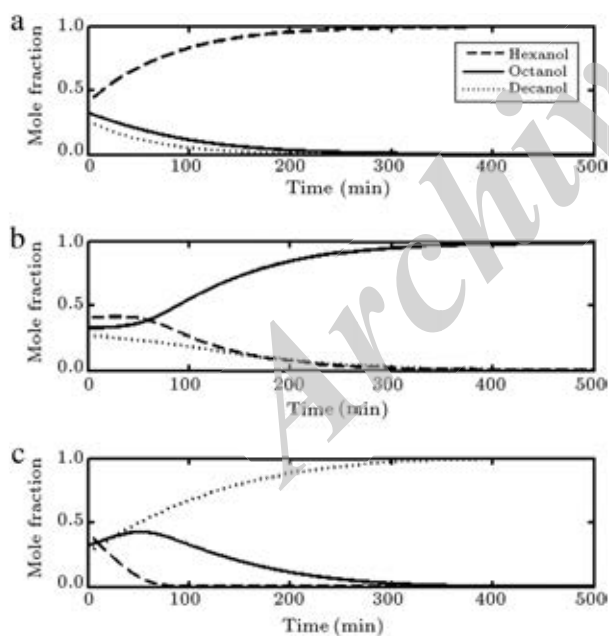


Figure 4: Compositions of conventional configuration. (a) Top; (b) middle; and (c) bottom.

the condensed vapor into the middle vessel appears as a time delay about 35 min.

In the modified system, the time needed for all products to achieve the final compositions which are at least 99% (mole fraction), will be about 101 min less than the conventional method. The reason of this difference is the middle vessel. Although the middle vessel in the modified configuration is an

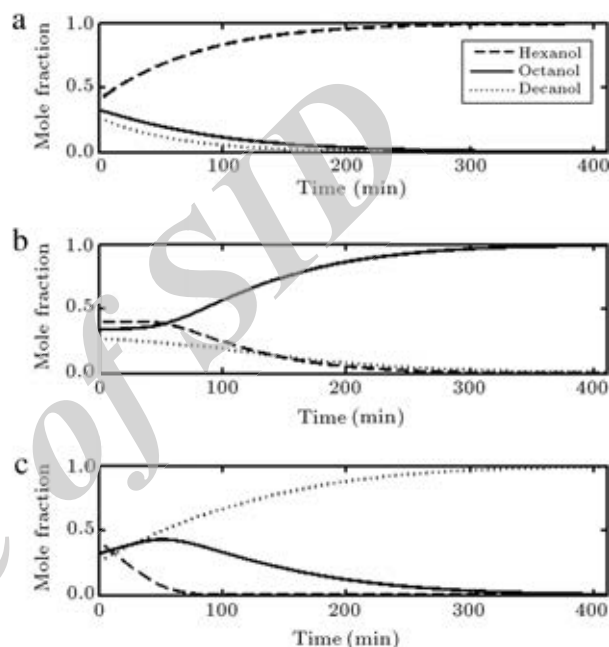


Figure 5: Compositions of modified configuration. (a) Top; (b) middle; and (c) bottom.

equilibrium stage like the other trays, it has much greater liquid holdup and the dynamic of the vessel has a large role in the separation and causes the time reduction.

For the two-cyclic column, the composition profiles for two vessels in each cycle are shown in Figure 6. In this configuration, the total time of separation is the sum of the time required for the first and the second cycle.

Operating costs of a distillation column mostly depend on the heat required for the reboiler and the heat removed in the condenser, so reboiler and condenser duties (Figure 7) are used to calculate the energy requirement. To compare the conventional, modified and two-cyclic batch columns, the total energy required in the reboiler and removed in the condenser and the operation time are shown in Table 6. As it can be seen, the modified configuration needs the least energy and time. Note that in all simulations, the operation is stopped when the main component of each vessel reaches its desired concentration which is at least 99% mole fraction as it is summarized in Table 6.

3.3. Column profiles

To know more about the changes in the columns, a few important graphs for the modified middle vessel configuration are gathered.

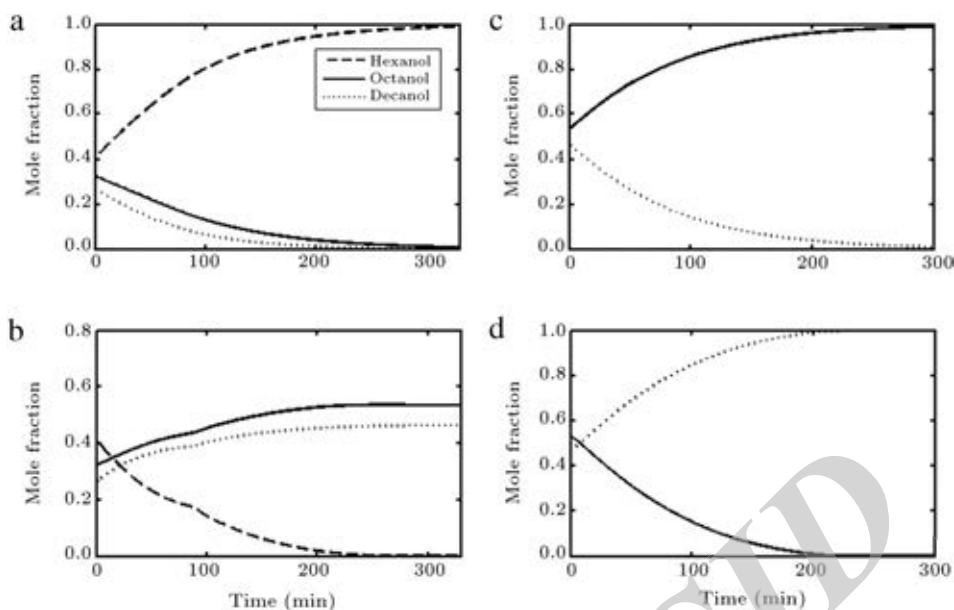


Figure 6: Compositions of rectifier configuration. (a) and (b) top and bottom of cycle 1; (c) and (d) top and bottom of cycle 2.

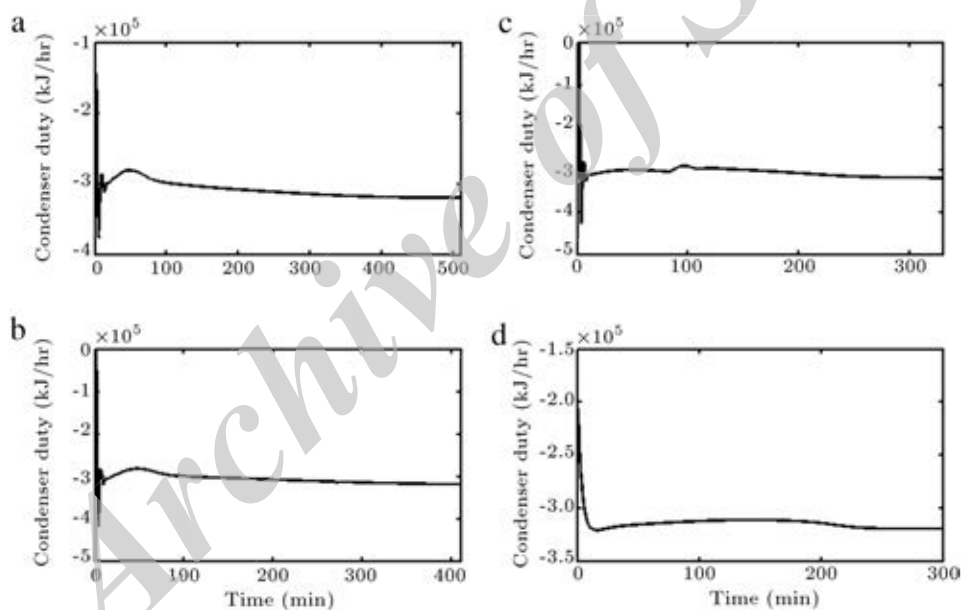


Figure 7: Condenser duty. (a) Conventional; (b) modified; (c) cycle 1; (d) cycle 2.

Figure 8 shows the temperature profiles for all of the trays in different time ranges. At the start of the process, reboiler starts to produce vapor and net vapor flow increases very fast. On the other hand, vapor condenses at the top vessel and flows to the column as the reflux and compositions change a lot from their initial values, so temperatures have large variations (Figure 8(a)). In Figure 8(b) temperature changes are little and steady profiles are seen in the column roughly, because the compositions do not have intensive variations. For example in tray 8 which has the most changes, it can be seen that the compositions have limited range of changes (Figure 9). Due to the fact that compositions change intensively during the time range of 200–410 min as it was mentioned in Figure 9, it is anticipated that the similar changes of temperature happen during this time period (Figure 8(c)).

Reboiler causes the most variation of the pressure in the bottom tray (21) but because pressure of the column is controlled by the condenser duty, bottom pressure reaches a steady state value after about 100 min as shown in Figure 10.

Net vapor profiles for two stages in rectifying and stripping sections are shown in Figure 11. These profiles show that a constant vapor molar flow is not a good assumption for this process.

As a result of dynamic process, liquid holdup in the columns differs during the time. Large liquid holdup can cause problems like flooding or loading. To the contrary, small holdup results dry packing regions. In Figure 12, it is shown that how the holdup of stages (different heights) changes in the column.

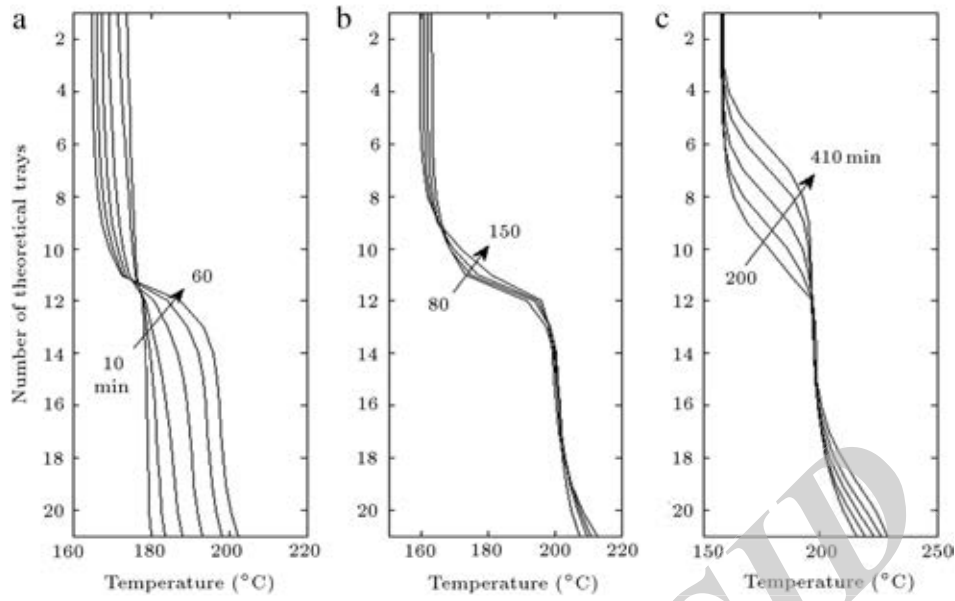


Figure 8: Temperature profile for modified configuration.

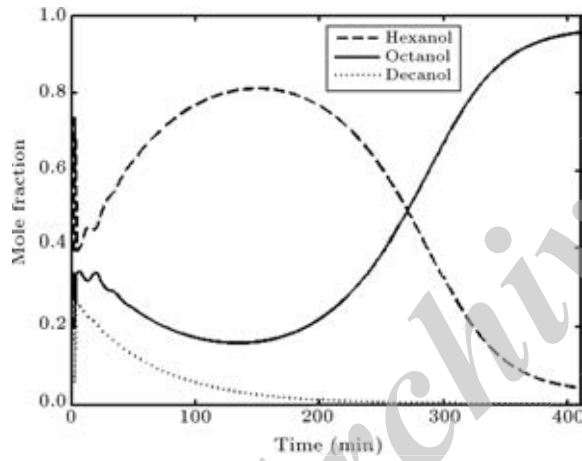


Figure 9: Composition profile of tray 8 in modified configuration.

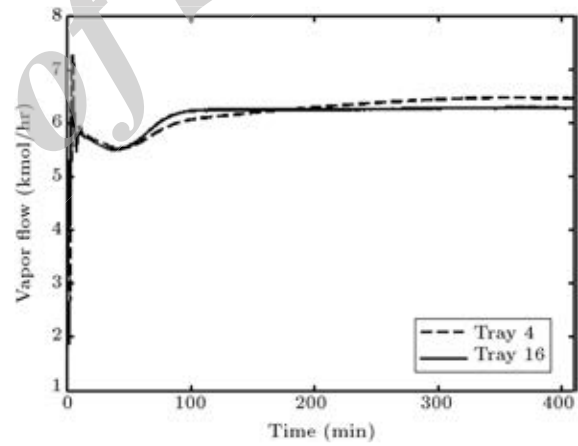


Figure 11: Vapor flow variation in modified configuration.

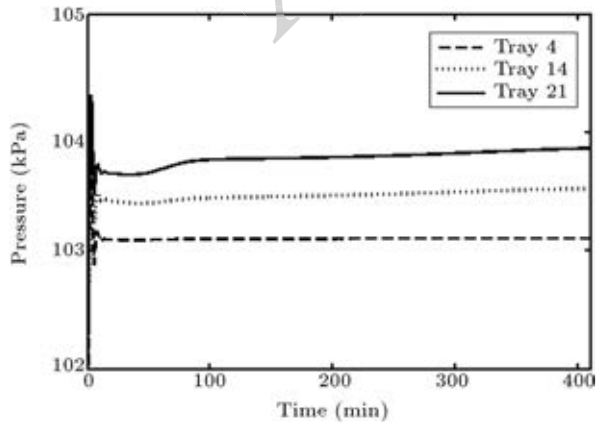


Figure 10: Pressure profile for specified trays of modified configuration.

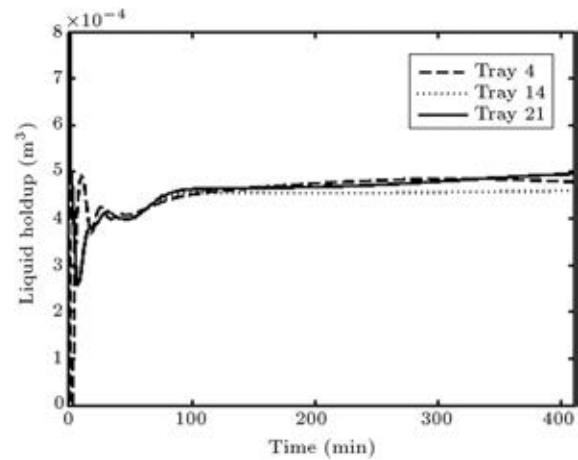


Figure 12: Holdup variation in different stages of modified configuration.

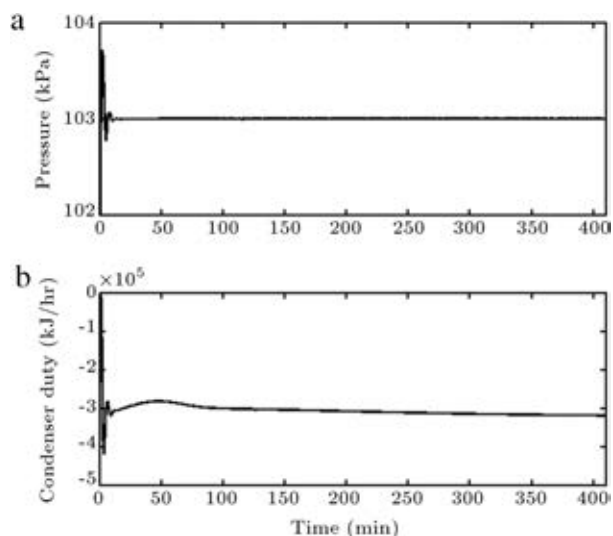


Figure 13: Transient response of pressure control loop. (a) Pressure; and (b) condenser duty.

3.4. Performance of level control structure

We know that a good controller must have some specifications. For example:

- Its output signal (*OP*) must have the lowest oscillations as possible.
- The Process Variable (*PV*) should reach its set point immediately.
- Process variable must not deviate from its set point during the operation.

The level control strategy has three control loops, one pressure and two level control loops.

To investigate the performance of these controllers, the controlled variable and the output of each controller for the modified middle vessel configuration are shown in Figures 13–15. As it can be seen from the results, the control structure has an acceptable performance and after about 80 min the process variables reaches their set points. Transient response of the bottom level is shown in Figure 16.

3.5. Temperature control versus the level control structure

As illustrated earlier, in the temperature control structure, reflux flows are used to control the temperature of the specified trays [9]. In this section, an extended temperature control structure is considered for controlling the modified middle vessel configuration. The selected structure has three controllers that control the column pressure by condenser duty, the temperature of fourth tray by reflux flow to rectifying section and the temperature of fifteenth tray by reflux flow to stripping section. The simulated flowsheet of the modified middle vessel column with selected temperature control structure is shown in Figure 17. In addition, the specifications of these controllers are shown in Table 7. It is necessary to mention that to prevent the operational instability, the top and middle temperature control loops start to act after about 98 and 68 min, respectively when the temperatures of the trays become close to their set points. In Figures 18 and 19, temperature changes of controlled stages and the way that reflux flows vary during the process are presented. The resulted transient responses of components in

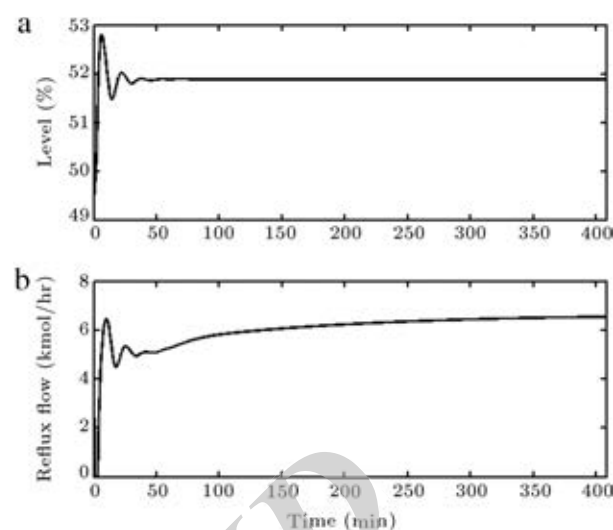


Figure 14: Transient response of top vessel level loop. (a) Level; and (b) reflux flow to rectifying section.

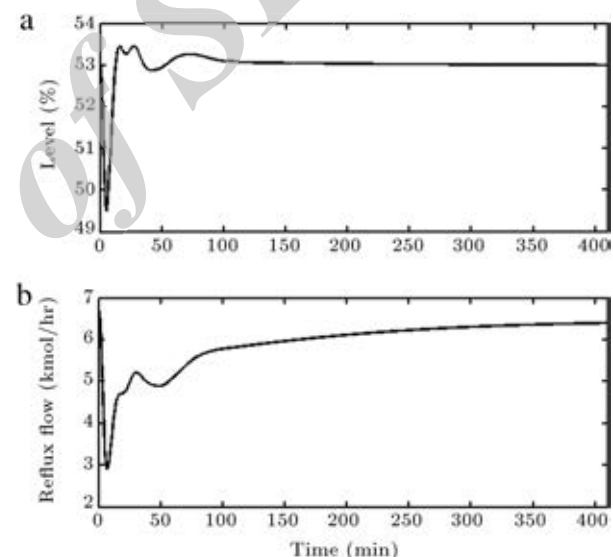


Figure 15: Transient response of middle vessel level loop. (a) Level; and (b) reflux flow to stripping section.

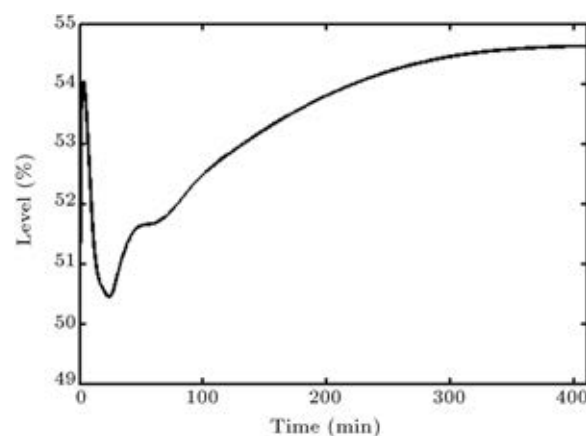


Figure 16: Transient response of bottom vessel level.

Table 7: Controllers parameters of the temperature control structure.

Controller	K	τ_i (min)	SP	PV_{\min}	PV_{\max}
Pressure ^a	9.28	0.44	103 kPa	80 kPa	120 kPa
Rectifying section ^b	0.2	1.5	186.7 °C	130 °C	210 °C
Stripping section ^b	0.5	1	202.6 °C	160 °C	240 °C

^a OP_{\min} and OP_{\max} for pressure controller are -155.56 and 0 kW, respectively.

^b OP_{\min} and OP_{\max} for these controllers are 0% and 100% , respectively.

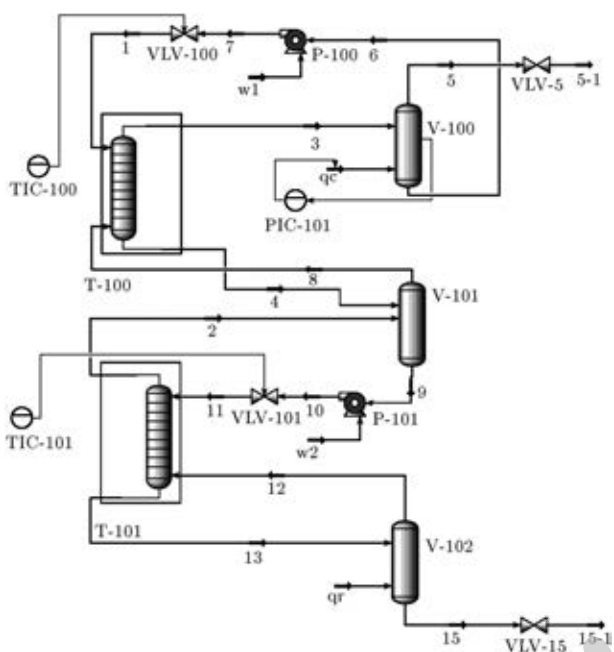


Figure 17: Simulated temperature control structure of modified middle vessel column.

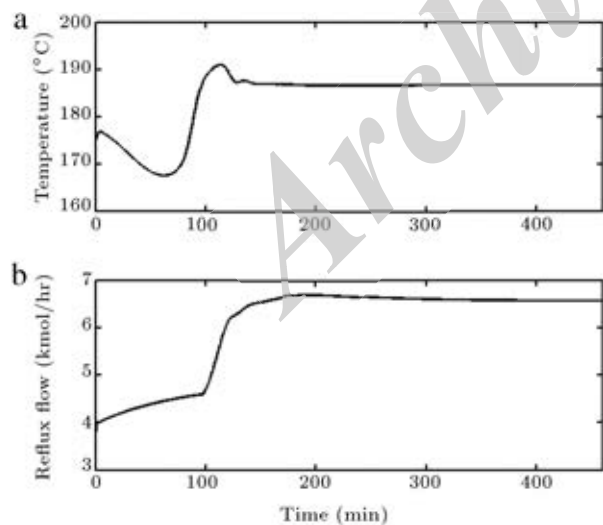


Figure 18: Transient response of top section temperature loop. (a) Temperature; and (b) reflux flow to rectifying section.

each vessel are shown in Figure 20. In comparison with level control structure, temperature control strategy needs more time and energy (Table 6).

In the temperature control structure, initially the controllers are off until the temperature of the trays which are chosen

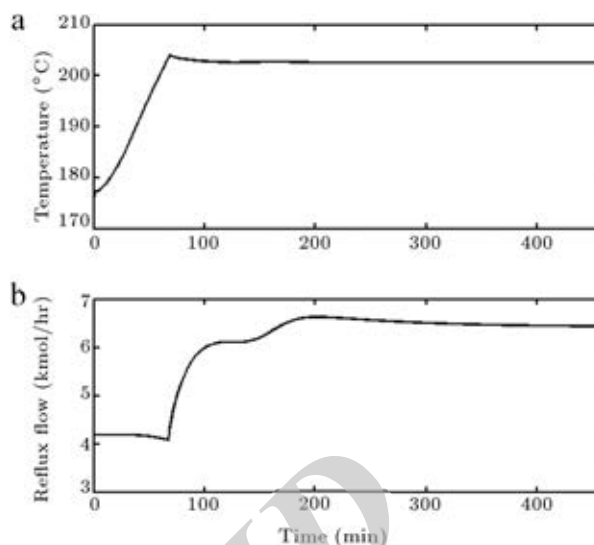


Figure 19: Transient response of bottom section temperature loop. (a) Temperature; and (b) reflux flow to stripping section.

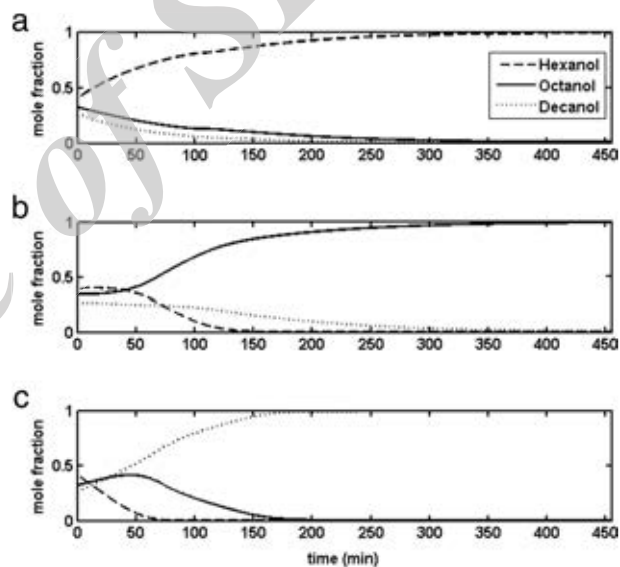


Figure 20: Compositions of modified configuration with temperature control strategy. (a) Top; (b) middle; and (c) bottom.

to control, becomes close to their set points; the process in this time range (startup) is highly nonlinear and therefore the controllers cannot work properly. This policy has a main disadvantage that the process is not controllable at the startup period. Furthermore it is too sensitive to the initial refluxes and needs to have some experience to know how much the reflux valves must be open to have the optimum operation. Figure 21 shows the level changes in the top and middle vessels for the simulated modified column when the temperature control structure is applied. In this figure, high changes of the levels are seen and they reach their desired values at the end of operation approximately; so the levels are controlled indirectly. In the level control structure, feed composition is the most important factor to determine the set points of the level controllers but in the temperature control structure, final temperature profile in the column which results in the appropriate compositions in the vessels are used. So it should be considered how these two controlling structures work when the feed composition

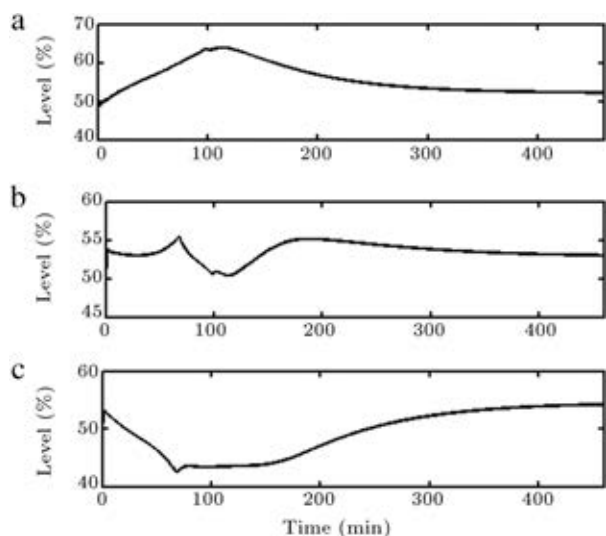


Figure 21: Level changes in modified configuration with temperature control structure. (a) Top vessel; (b) middle vessel; and (c) bottom vessel.

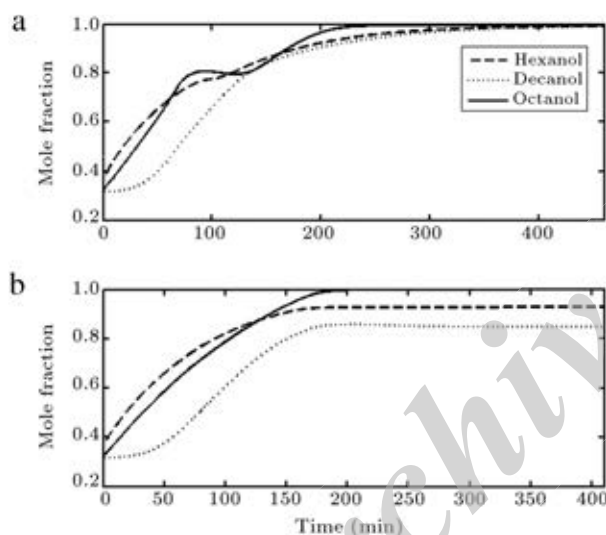


Figure 22: Compositions of main components with applying uncertainty in the feed. (a) Temperature control structure; and (b) level control structure.

is uncertain. If we change the initial feed concentrations from equal mass fractions to [0.3, 0.3 and 0.4] without changing the set points, level control structure is unable to do the separation completely and the final mole fraction of decanol would be about 83%, but temperature control strategy works properly as it can be seen in Figure 22.

4. Conclusion

In this paper, dynamic simulation and control of batch distillation columns were investigated. For this purpose, three configurations for separating of a tertiary zeotropic mixture were selected. In two configurations, the column has two sections with three accumulation vessels (top, middle and bottom), namely, conventional and modified middle vessel column. The third configuration is a rectifier column with two vessels (top and bottom) that is used in two cycles for separating these components. Unlike the previous papers, a

detailed model without assumptions of constant pressure and boilup flow was used. This model was simulated in Aspen Hysys 2009 software. Results have shown that the modified configuration has the lowest energy and time requirement. In addition, a control structure which has one pressure and one/two level controllers was selected for each configuration. Also the results have shown that the selected control structure has acceptable performance for three configurations and the process is more controllable with this strategy than the temperature control structure especially at startup period and causes to reduce the time and energy needed. It is necessary to mention that when the feed composition is uncertain, unlike the temperature control structure, the products cannot be reached the desired values by using the level control strategy.

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