Brazilian Journal
of Chemical
Engineering

Vol. 35, No. 02, pp. 769 - 784, April - June, 2018
dx.doi.org/10.1590/0104-6632.20180352s20160522

ISSN 0104-6632
Printed in Brazil
www.scielo.br/bjce

OPERATIONAL DESIGN AND IMPROVEMENT OF CONVENTIONAL BATCH DISTILLATION AND MIDDLE-VESSEL BATCH DISTILLATION

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(Submitted: September 2, 2016; Revised: March 11, 2017; Accepted: April 10, 2017)

Abstract - Conventional batch distillation and middle-vessel batch distillation were studied for the separation of the ternary system of cyclohexane/n-heptane/toluene, which has low relative volatilities. The modified variable and constant reflux operations were applied in the conventional batch distillation. Two control structures were used: one of which was referred to as the composition control structure with a modified level-set point, and the other was referred to as the flowrate-limiting control structure. Thus, the middle-vessel batch distillation was able to simultaneously separate the components of the ternary mixture. Comparing the two control structures showed that the flowrate-limiting control structure was better than the composition control structure with a modified level-set point in terms of their applicability. Disturbances were introduced to assess the controllability of the flowrate-limiting control structure. The dynamic responses demonstrated that the control structure performed well. The results showed that the modified conventional batch distillation and middle-vessel batch distillation increased the purity of the separated components compared to previous studies.

Keywords: Batch distillation, Middle vessel, Control structure, Ternary system.

INTRODUCTION

Batch distillation is widely used in the pharmaceutical industry to purify and recover high-value liquid components. It has better operational flexibility and reduces equipment costs compared to continuous distillation (Jimenez *et al.*, 2002; Narvaes-Garcia *et al.*, 2015). Over many years of improvement, batch distillation processes have been developed that can be used in special distillation processes, such as pressure-swing distillation (Modla, 2011; Repke *et al.*, 2007), extractive distillation (Fang *et al.*, 2010; Navarrete-Contreras *et al.*, 2014; Pacheco-Basulto *et al.*, 2012), and reactive distillation (Khazraee and Jahanmiri, 2010; Modla, 2011), which are based on different column configurations, including conventional and unconventional column configurations.

Conventional batch distillation can be operated in several modes, and the performance of the operation modes is different. Many researchers (Bai *et al.*, 2007; García *et al.*, 2014; Lopes and Song, 2010) have studied operation optimization and made contributions to the process. However, the column

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cannot be used to separate components simultaneously when the mixtures contain more than two components.

Unconventional column configurations, including inverted batch distillation columns, middle-vessel batch distillation (MVBD) columns, and multivessel batch distillation columns, have all been studied in order to solve more complex situations, such as multicomponent separation (Mujtaba, 2004).

MVBD columns have gained considerable attention because of their great flexibility (Gruetzmann *et al.*, 2006; Leipold *et al.*, 2009; Warter *et al.*, 2004). The middle vessel between the rectifying and stripping sections can act as a feed tank and product vessel. In the case of a separation of a ternary mixture, for instance, the lightest/heaviest components are collected at the top/bottom, while the intermediate component is purified in the vessel simultaneously. Additionally, the vessel can be used as an additional reboiler or condenser so that one more degree of freedom is added, thus making the process more flexible. Quintero-Marmol and Luyben (1990) have explored the operation modes of multicomponent batch distillation columns. One of them, known as fed-batch distillation, involves a setup with two middle vessels, which results in a higher capacity factor than in the regular mode. Davidyan *et al.* (1994) theoretically analyzed the dynamic behavior of MVBD and revealed the influence of the parameters. This paper has widely prompted the development of MVBD. Meski and Morari (1995) solved the optimal control problem of MVBD in the case of a binary separation and found that the minimum reflux model was the best suited control policy. Barolo *et al.* (1996) carried out experimental runs in a pilot plant-scale continuous distillation column that had been modified to implement batch operations. Then, Barolo *et al.* (1998) developed a detailed model to investigate the effect of the operating and design parameters. Warter *et al.* (2002) compared the experimental and simulation results of separating a ternary zeotropic system in a cyclic operation with a constant holdup, and they explained the limits of the geometrical and operational parameters. Diwekar (2011) comprehensively analyzed the behavior of the process in a shortcut model, rigorous model and semi-rigorous model. Rao and Barik (2012) studied the effects of reboiler heat duty, reflux flowrate and number of plates on the product composition and dealt with the control of the distillate composition and condenser holdup. In addition, the studies of MVBD columns have been extended to several special distillation processes. Cheong and Barton (1999a, 1999b, 1999c) used the concept of warped time analysis to develop a mathematical model and studied the qualitative dynamics of MVBD for the separation of azeotropic mixtures in a case where the separation boundaries were linear and nonlinear. Warter and Stichlmair (2000) described three MVBD processes for the separation of a binary azeotropic mixture using an entrainer and found that, when the liquid from the upper section was sent to the lower section directly, the process performed best. Low and Sørensen (2002) investigated the optimal operations of MVBD columns used in extractive distillation based on a profit function. Control strategies are essential for the process to achieve high-purity products. Phimister and Seider (2000a, 2000b) studied two distillate-bottom (DB) control configurations and overcame the associated deficiencies, such as low product purity. Gruetzmann and Fieg (2008) analyzed and discussed the initiation of an MVBD column and used a temperature control structure to investigate the process. Fanaei *et al.* (2012) compared the control structures of conventional middle-vessel batch distillation and modified middle-vessel batch distillation, and they found that level controllers could obtain high purity products when the feed composition was kept constant. Luyben (2015) studied the control structures of MVBD for separating a ternary system, and the results showed that a composition control structure and temperature control structure could perform well for controlling the product purity and liquid holdup. Zhu *et al.* (2016) compared the composition control structure and two temperature control structures and found that the temperature control structures performed better.

In this paper, we studied the conventional batch distillation process and MVBD process for the separation of a ternary system of cyclohexane/n-heptane/toluene, which is difficult to separate because of their low relative volatilities. More efficient operations in the conventional batch distillation were adopted, and new control structures in the MVBD were explored to stabilize the product purity and liquid holdup. Both distillation processes could effectively separate the ternary mixture into high-purity products.

MODIFIED OPERATIONS OF CONVENTIONAL BATCH DISTILLATION

Conventional batch distillation for the separation of the mixture of cyclohexane/n-heptane/toluene was simulated by Aspen Batch Distillation with the SRK model and was operated under two modes: variable reflux and constant reflux. The number of stages was set at 41, which was large enough to separate the ternary system through conventional batch distillation (Figure 1a) and the MVBD (Figure 1b).

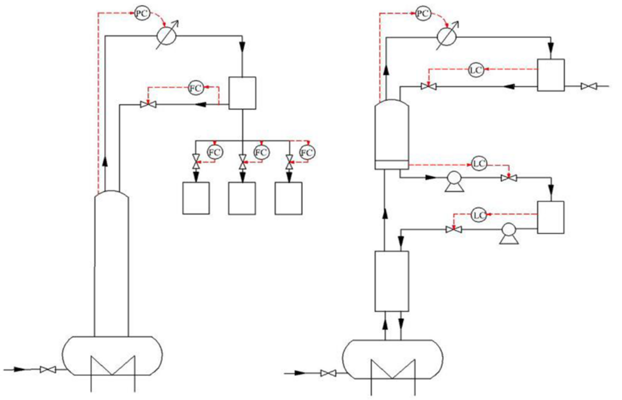

Figure 1. The flowsheets of: (a) conventional batch distillation; and (b) middle-vessel batch distillation.

Modified variable reflux operation

A modified variable reflux operation was used to maintain the component purity in the corresponding product vessel at a desired value by changing the reflux ratio. The liquid holdup of the feed in the bottom was set at 1000 kmol with a composition of 40.7 mol% cyclohexane, 39.4 mol% n-heptane, and 19.9 mol% toluene (Jain *et al.*, 2012). Three product receivers were connected with the distillate stream to collect the main-cuts and off-cuts. Receiver 1 was used to collect high-purity cyclohexane at step 1, and receiver 3 was employed to gather high-purity n-heptane at step 3. On the other hand, receiver 2 was used to collect a mixture of cyclohexane and n-heptane that did not meet the purity demands of the products at step 2. The column was operated at atmospheric pressure, and the modified operation steps are shown in Table 1.

The medium heating temperature of the setup was based on the temperature of 5-atm saturated steam (absolute pressure), which has a sufficient difference in temperature from the boiling points of the mixture. In the modified steps, the distillate mole flowrate in each step had an impact on the product purities and liquid holdups. Figure 2 shows the effects of distillate flowrate on the key factors of each step.

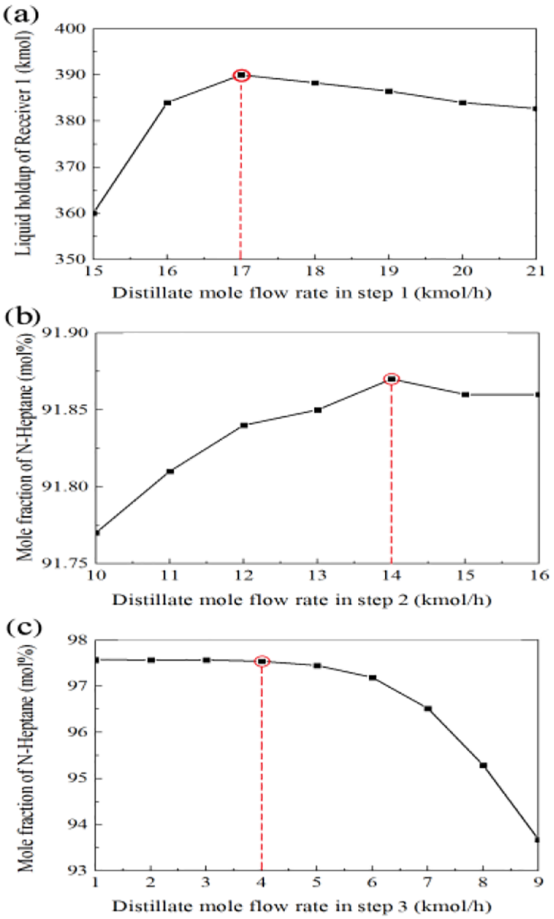

Figure 2. Effect of distillate mole flow: (a) on Receiver 1 liquid holdup in step 1; (b) on n-heptane mole fraction in step 2; and (c) on n-heptane mole fraction in step 3.

It should be noted that the distillate flowrate in step 3 slightly influenced the mole fraction of n-heptane in the third receiver when the rate was less than 4 kmol/h. Increasing the distillate flowrate could decrease the batch time; therefore, 4 kmol/h was selected as one of the operating parameters. The mole fraction (X) and liquid holdup (M) in the three product vessels and the reflux ratio are shown in Figure 3 and Table 2.

The modified variable reflux operation could achieve higher purity of the three products, especially for cyclohexane and n-heptane, compared to the results of a previous study (Jain *et al.*, 2012), which reported product purities of 89.3 mol% cyclohexane, 86.3 mol% n-heptane, and 99.1 mol% toluene.

Table 1. Modified steps of the variable reflux operation.

Set Variables	Step 1	Step 2	Step 3
F_d (kmol/h)	17	14	4
T_{hm} (°C)	150	150	150
Sequence of receivers	No. 1	No. 2	No. 3
End conditions	$X_{receiver1, cyc}$=99 mol%	$X_{pot, cyc}$=1 mol%	$X_{pot, tol}$=99 mol%

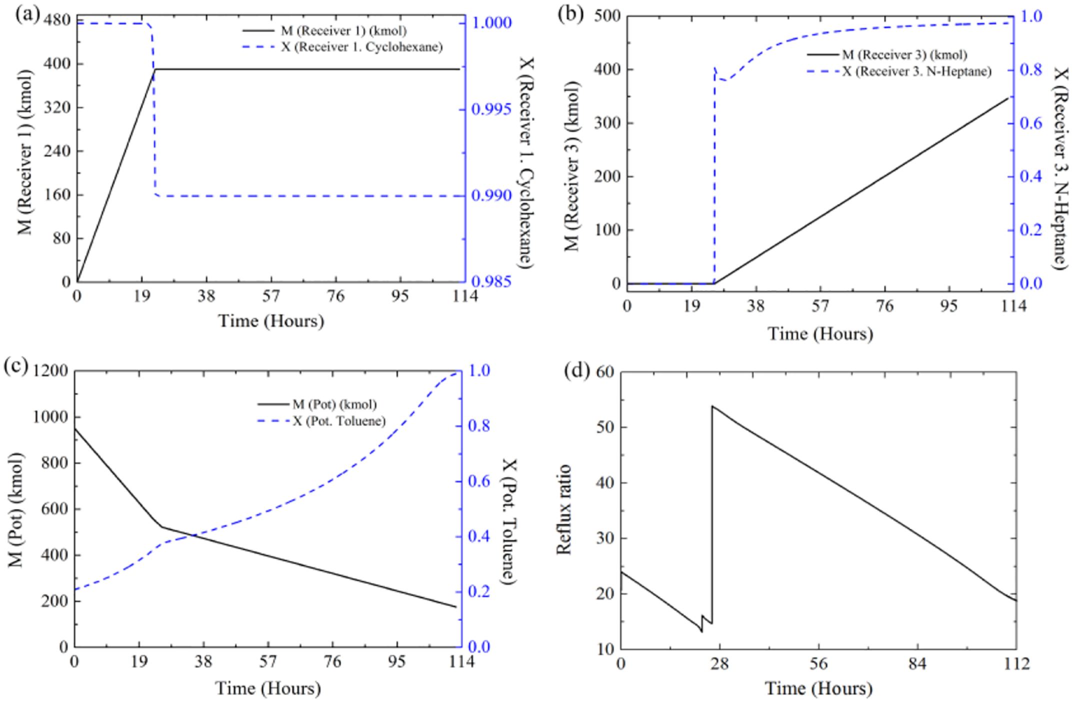

Figure 3. Variation trends of: (a), (b), (c) product purity and liquid holdup; and (d) reflux ratio.

Table 2. Results of the modified variable reflux operation.

Parameters	Receiver 1	Receiver 2	Receiver 3	Pot
X_{cyclo} (mol%)	99.0	33.5	2.3	trace
$X_{n\text{-}hep}$ (mol%)	1.0	66.4	97.5	1.0
X_{tol} (mol%)	trace	0.1	0.2	99.0
M (kmol)	390.1	38.9	346.2	175.7

Modified constant reflux operation

The modified constant reflux operation allowed the achievement of products with the desired purity by fixing the reflux ratio. The initial settings in this operation were the same as the modified variable reflux operation, and three vessels were used to collect the products and byproducts. The reflux ratio was set to 15 because the mole fraction of n-heptane was already higher than 99.0 mol% and remained almost constant with the increase of the reflux ratio (Figure 4).

The modified operation steps are shown as Table 3.

The end condition of the second step was that the overall holdup of the receiver reached 140.0 kmol because the setting had positive impact on the n-heptane quantity and purity (>99.0 mol%). The results of the constant reflux operation are shown in Table 4 and Figure 5.

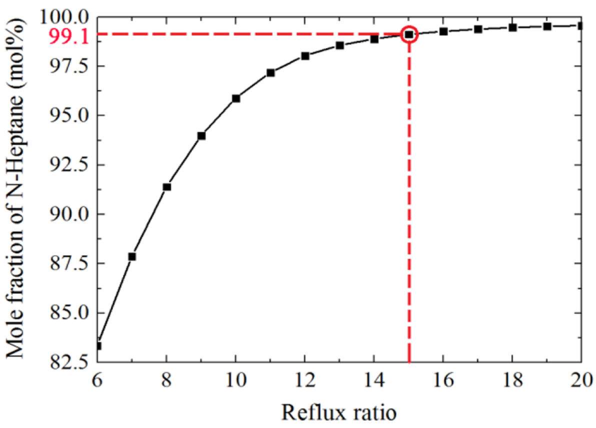

Figure 4. Mole fraction of n-heptane under different reflux ratios.

The two operation modes of conventional batch distillation separated the ternary system of cyclohexane/n-heptane/toluene efficiently, and the purities of cyclohexane and n-heptane were improved. However, the conventional batch process could not separate the components simultaneously. As an important unconventional batch distillation process, MVBD could overcome the bottleneck as a result of effective control structures.

Table 3. Modified steps of the constant reflux operation.

Set Variables	Step1	Step2	Step3
Reflux ratio	15	15	15
T_{hm} (°C)	150	150	150
Sequence of receivers	No.1	No.2	No.3
End conditions	$X_{receiver1, cyc}$=99 mol%	$M_{receiver2}$=140 kmol	$X_{pot, tol}$=99 mol%

Table 4. Results of the modified constant reflux operation.

Parameters	Receiver 1	Receiver 2	Receiver 3	Pot
X_{cyclo} (mol%)	99.0	14.4	0.2	trace
$X_{n\text{-}hep}$ (mol%)	1.0	85.4	99.1	1.0
X_{tol} (mol%)	trace	0.2	0.7	99.0
M (kmol)	390.2	140	253.7	167.1

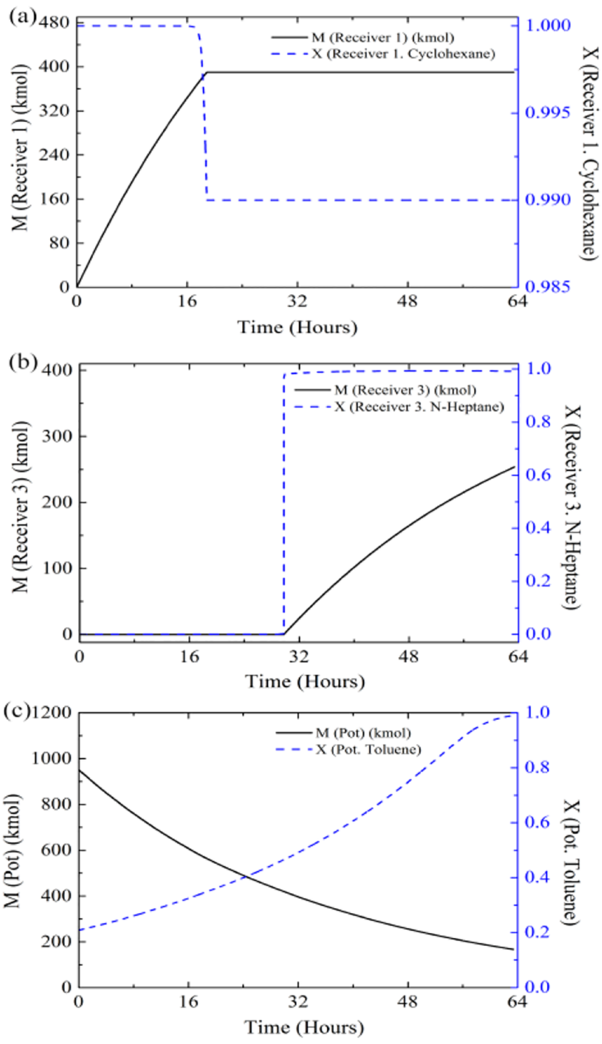

Figure 5. Variation trends of product purity and liquid holdup.

DESIGN AND CONTROL OF MIDDLE-VESSEL BATCH DISTILLATION

MVBD, as a typical model of unconventional batch distillation, was studied using Aspen Plus and Aspen Plus Dynamics in this section. The SRK physical properties were selected, and the basic setup procedure was similar to that proposed by Luyben (2015), although the control structures were different.

Process setup in steady-state Aspen Plus

The MVBD column was simulated by two *Radfrac* models, with one being used as the rectifying section and the other as the stripping section. In addition, a *Flash 2* model was used to simulate the middle vessel. Several valves and pumps were added to change the pressure of the streams. More details about the simulation are shown in Table 5.

The flowsheet of the MVBD is shown in Figure 6, and the feed stream was set at 1000 kmol/h with a composition of 40.7 mol% cyclohexane, 39.4 mol% n-heptane, and 19.9 mol% toluene. The column and middle vessel were operated at atmospheric pressure. The size of each vessel needed to be large enough to accommodate the components based on the feed volume. The process could not be defined completely as a batch distillation process because there were several feed streams. Some adjustments were carried out to implement the batch process in a dynamic simulation.

The results of the steady-state processes show that there was almost 100 mol% cyclohexane in the reflux drum and 99 mol% cyclohexane in the middle vessel. A mixture of 41.0 mol% cyclohexane, 39.0 mol% n-heptane, and 20.0 mol% toluene remained in the sump (Table 6). These results mean that MVBD is unlike continuous distillation because the separation of the mixture in MVBD required an efficient control structure to implement.

Table 5. Simulation details in steady-state Aspen Plus.

Upper Section (US)

Stage number: 20 (includes the condenser)

Section diameter: 0.88 m (calculated by tray sizing)

Reflux drum: length 3.5 m, diameter 4 m (used to collect cyclohexane)

Sump: length 0.8 m, diameter 2 m

Distillate flowrate: 1 kmol/h

Lower Section (LS)

Stage number: 21 (includes the reboiler)

Section diameter: 0.84 m (calculated by tray sizing)

Sump: height 5.5 m, diameter 6 m (used as the feed tank and toluene product vessel)

Distillate flowrate: 100 kmol/h

Middle vessel (MIDDLE-V)

Pressure: 1 atm

Dimension: length 4.6 m, diameter 4 m (used to collect n-heptane)

Valves

VD2, VUPPER: outlet pressure 1.1292 atm (equal to the pressure at the bottom of the US column)

VF: outlet pressure 1.272 atm (equal to the pressure at the bottom of the LS column)

VMID: outlet pressure 1.136 atm (equal to the pressure at the top of the LS column)

VD, VVENT: outlet pressure 0.9 atm

VUP, VB: outlet pressure 1 atm

Pumps

PU, PMID: pressure increase 10 atm

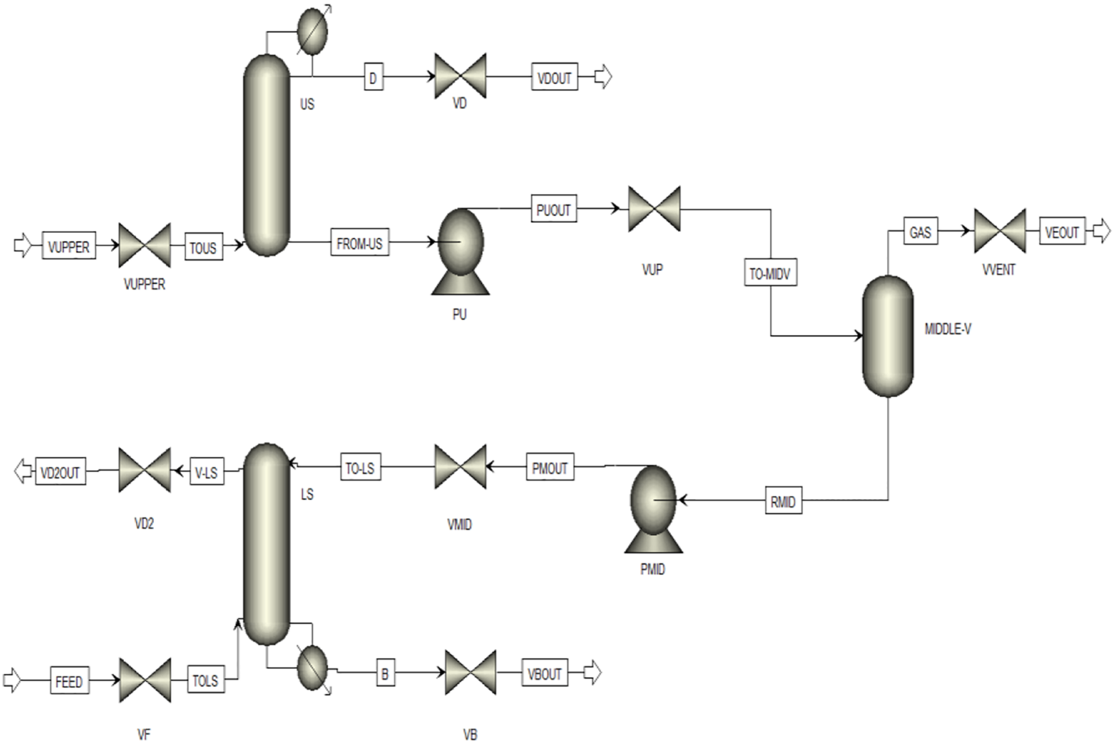

Figure 6. The flowsheet of the middle-vessel batch distillation.

Table 6. Stream results of the steady-state simulation.

Parameters	D	RMID	B	GAS	TO-LS	FEED	VUPPER	FROM-US
F_{total} (kmol/h)	1.00	98.99	998.99	0.001	98.99	1000.00	100.00	99.00
F_{cyclo} (kmol/h)	1.00	97.99	405.70	0.001	97.99	407.00	99.00	98.00
$F_{n\text{-}hep}$ (kmol/h)	Trace	1.00	394.29	Trace	1.00	394.00	1.00	0.01
F_{tol} (kmol/h)	-	-	199.00	-	-	199.00	-	-
X_{cyclo}	1.00	0.99	0.40	0.99	0.99	0.41	0.99	0.99
$X_{n\text{-}hep}$	8PPM	0.01	0.40	0.01	0.01	0.39	0.01	0.01
X_{tol}	-	-	0.20	-	-	0.20	-	-

Level control structure with a temperature controller

In the dynamic mode, the valves VD, VVENT, VF, and VB were closed by setting their openings to zero. The streams VUPPER and VD2OUT, and the valve VUPPER were deleted when the stream TOUS was connected to the valve VD2. Therefore, the system became a closed system, and it could be defined as a batch distillation process.

The mixture was fed into the sump of the LS column, then the sump was heated. The operating pressure of the process was 1 atm. With the temperature increase, almost all the light component (cyclohexane) and a certain amount of the intermediate component (n-heptane) were vaporized and moved up into the condenser at the top of the column. Part of the condensed mixture was refluxed to ensure the high purity of the light component in the reflux drum; thus, the reflux flowrate was important for the cyclohexane content. Then, the mixture was refluxed back to the column, and liquid holdup was established in the trays and in the middle vessel. The sump of the US column held the liquid to be pumped into the middle vessel. As the temperature of the sump in the LS column increased further, the composition of n-heptane in the middle vessel was profoundly influenced by the toluene that boiled up into the US column. As a result, the heat duty of the reboiler and the reflux flowrate to the LS column should be controlled strictly. When the process became steady, the components should be collected with high purity in the reflux drum, middle vessel and sump of LS column. The composition, level, and flowrate were correlated with each other, and the control structures needed to be set to achieve effective separation.

Level control structures (LCS) have been studied in batch distillation (Fanaei *et al.*, 2012; Luyben, 2015) and continuous distillation processes, such as pressure swing distillation (Wei *et al.*, 2013; Zhu *et al.*, 2015), reactive distillation (Xu *et al.*, 2014), and extractive distillation (Wang *et al.*, 2015). In this paper, three level controllers named LCtop, LCbase, and LCmid, a temperature controller called TCLS and a pressure controller called PC were added to stabilize the separation system. The detailed control structure is as follows:

1. The heat duty of the condenser in the column was manipulated (reverse acting) to control the operating pressure of the column.
2. The flowrate at the top (direct acting) was manipulated to control the level of the reflux drum in the US column.
3. The flowrate at the bottom (direct acting) was manipulated to control the base level in the US column.
4. The flowrate (direct acting) was manipulated to control the level in the middle vessel.
5. The heat duty of the reboiler in the column (reverse acting) was manipulated to control the temperature of stage 1 so that the toluene content that boiled up into the US column could be hindered.

It should be noted that the action of the PC is set as reverse for the value of the condenser duty is negative in the Aspen Dynamics. The flowsheet of LCS with a temperature controller is shown in Figure 7. The results demonstrated that the purity of cyclohexane and toluene was larger than 99.5 mol%, while n-heptane was only 71.0 mol%. In terms of the liquid holdup in the three product vessels, there were only 20.5 kmol in the reflux drum, 32.6 kmol in the middle vessel, and 84.5 kmol in the sump (Figure 8). Hence, it was important to explore a control structure to improve the product purity.

Composition control structure

Composition control structures (CCS) often perform better than LCS for improving product purity. A composition controller named CCtop, which was used to control the n-heptane content in the reflux drum by manipulating the reflux flowrate, and a high selector named HStop, which was a *MultiHiLoSelect* model, were set in the flowsheet (Figure 9). When

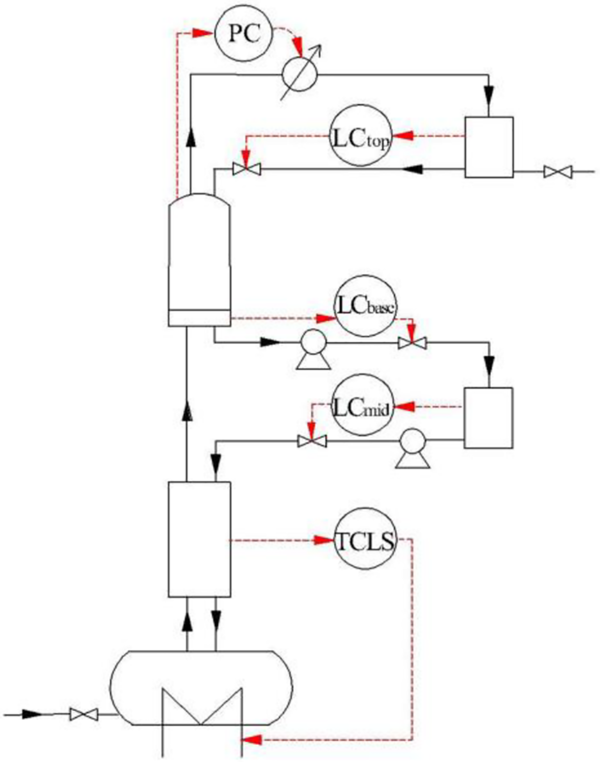

Figure 7. The flowsheet of the level control structure with a temperature controller.

the mole fraction of n-heptane in the reflux drum was higher than the set point, the reflux flowrate would be increased by the direct action of CCtop. HStop was used to select the larger value between "Input_1" and "Input_2" as the output signal and then to control the reflux flowrate of the upper section column. "Input_1" is a fixed value (8626 kg/h) that was almost equal to the initial value of the reflux flowrate, and the "Input_2" was the CCtop controller output signal. The parameters of controllers are significant for the performance, and there are differences between the tuning methods used in continuous distillation and batch distillation. Several tuning methods could be used in distillation processes, such as the Tyreus-Luyben method (Tyreus and Luyben, 1992), IMC method (Rivera *et al.*, 1986), and SIMC method (Skogestad, 2003, 2006), and they each have their own advantages when used under different conditions. For continuous distillation, we applied the Tyreus-Luyben tuning rule to calculate the gain and integral time when the process became steady-state. However, for batch distillation, especially for MVBD, the process variables were changing; thus, the Tyreus-Luyben tuning rule could not be used at the beginning. To solve this problem, the gain and integral

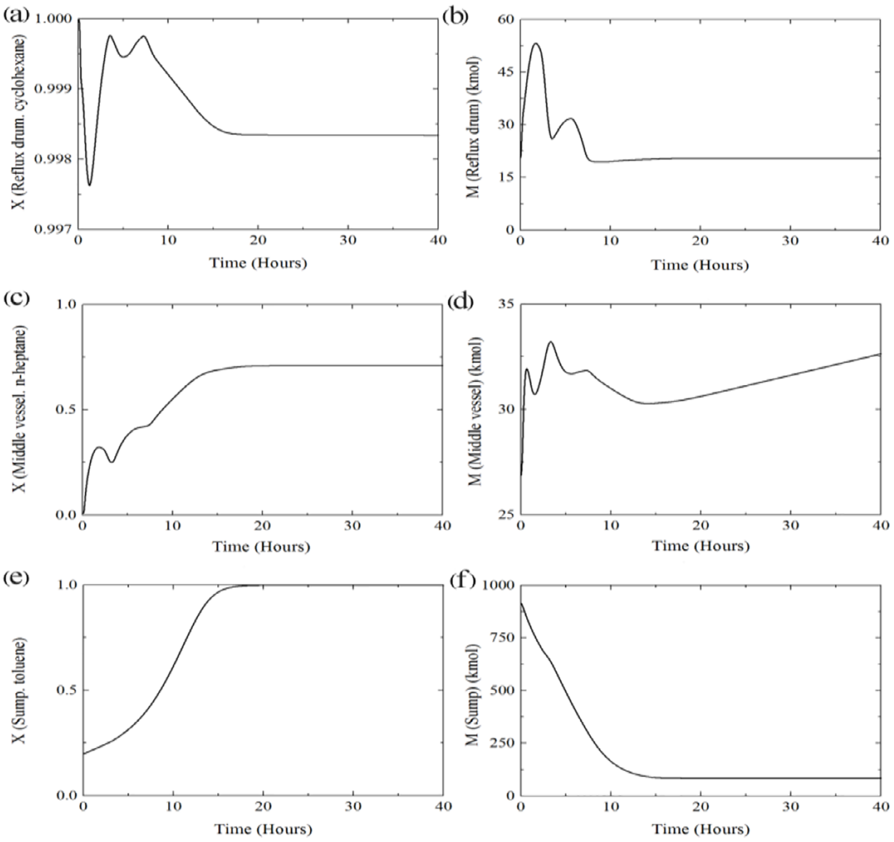

Figure 8. Results of the level control structure with a temperature controller.

time of the controllers were set empirically until the purities of products were stable at a high level, then the Tyreus-Luyben tuning was initiated to calculate the more appropriate gain and integral time. The gain and integral time of the composition controller CCtop were set to 5 and 20 min empirically, while those of the temperature controller TCLS were set to 1 and 20 min. When the product purities were high and constant, the Tyreus-Luyben tuning method was applied to calculate the more appropriate gains and integral times for the two controllers. Table 7 shows the parameters of each controller in the CCS.

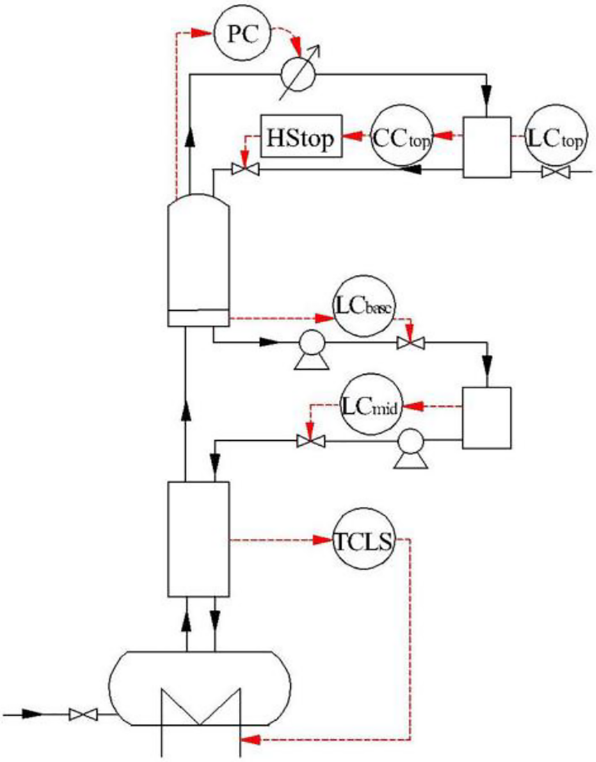

Figure 9. The flowsheet of the composition control structure.

Figure 10 shows that the cyclohexane purity decreased from 99.9 mol% to 99.0 mol% because a small amount of n-heptane entered into the reflux drum, while the toluene content continuously increased to 99.3 mol% in the sump as the light and the intermediate components boiled up. In terms of the n-heptane content, it was difficult to end up at 99.0 mol% due to the low relative volatility of n-heptane/toluene, and a certain amount of toluene was easily delivered into the middle vessel with n-heptane. The final liquid holdup was 297.9 kmol in the reflux drum and 117.2 kmol in the sump. However, it was only 29.0 kmol in the middle vessel because the flowrate of stream RMID was approximately equal to that of stream TO-MIDV (Figure 11a). To have a high liquid holdup in the product vessels, the flowrate of output streams needed to be smaller than that of input streams so that it was possible to assemble the amounts of the liquid products (Figure 11b and 11c).

Composition control structure with a modified level-set point

In the composition control structure, the level of the middle vessel, which could reflect the liquid holdup, was manipulated by LCmid, and the set point of the controller was crucial to this level. Therefore, the level of the middle vessel could be controlled to increase the liquid holdup, i.e., to indirectly decrease the flowrate of the output stream RMID. However, it should be noted that the flowrate from the vessel represented the reflux flowrate to the LS column. The decrease of the reflux flowrate could cause more toluene to boil up into the US column, which would lower the purity of n-heptane in the middle vessel. The relationship between the level in the middle vessel, n-heptane concentration, and liquid holdup of the middle vessel were investigated to determine an optimized value of the set point in LCmid to ensure that the liquid holdup was as high as possible while keeping the n-heptane content at 92.0 mol%. Figure 12 shows the effects of the level in the middle vessel on the n-heptane mole fraction and liquid holdup. When the level was 1.5 m, the composition of n-heptane was 92.0 mol%, and the liquid holdup was the highest. Therefore, the set point of level controller LCmid was set at 1.5. Table 8 gives the results of the composition control structure with and without the modified set point of LCmid. It was clear that the liquid holdup of the middle vessel was increased by 119.3 kmol, which meant that indirectly decreasing the flowrate of the output stream RMID by controlling the level of the middle vessel was feasible. However, Figure 11b shows that the flowrate of the output stream decreased immediately to zero at 1 h and then rose up. The high liquid holdup of the middle

Table 7. The tuning parameters of the controllers in the composition control structure.

Parameters	PC	CCtop	TCLS	LCbase	LCmid
gain K_c	20	6.642	124.264	1	1
integral time τ_I (min)	12	50.16	2.64	20	20

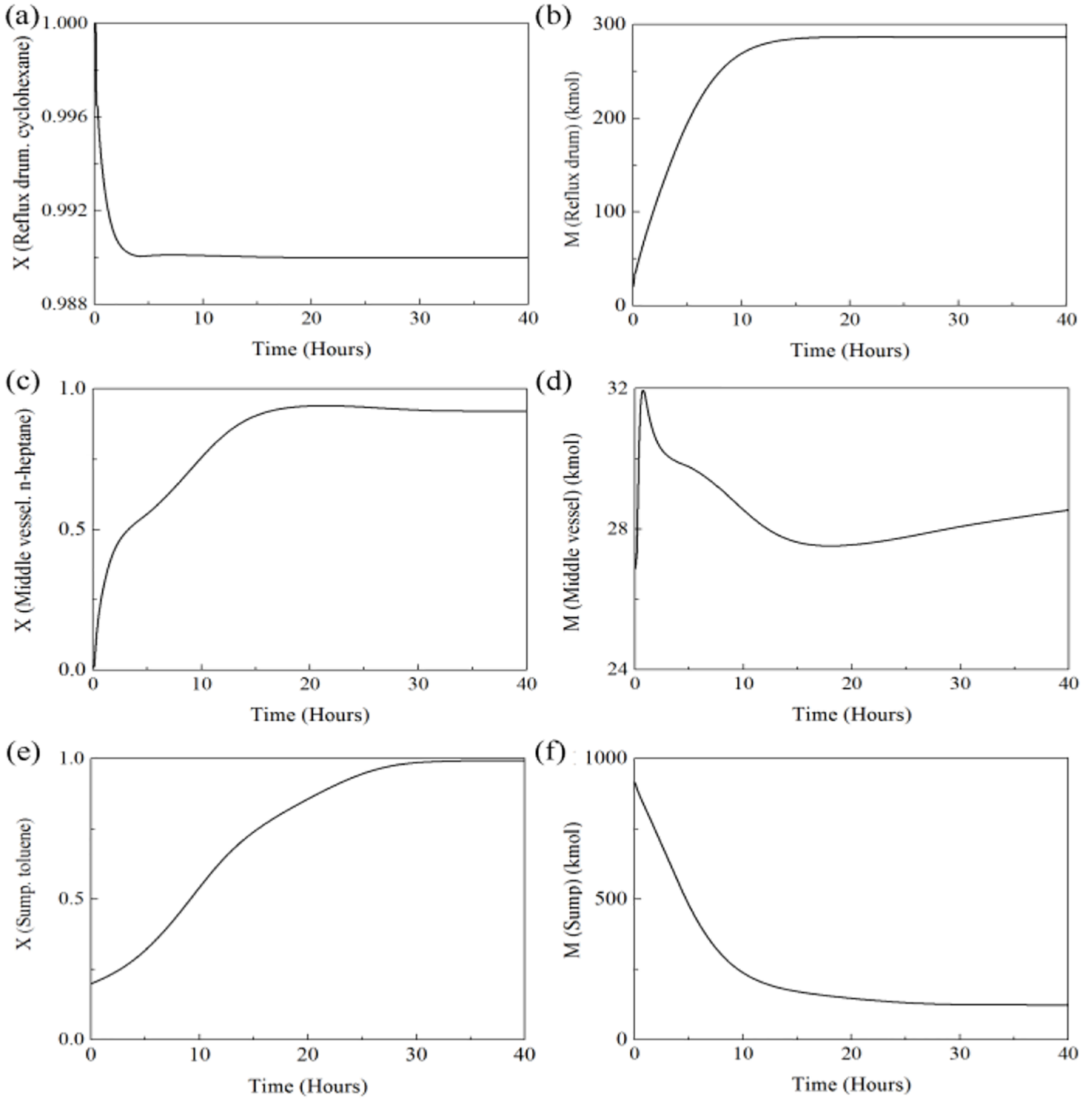

Figure 10. The results in terms of product purity and liquid holdup in each vessel.

vessel was formed during the first 3 h, then it changed slightly in terms of quantity, while the composition changed gradually.

Flowrate-limiting control structure

The flowrates of the streams are usually controlled by flowrate controllers in the continuous and batch distillation processes. To decrease the flowrate of the output stream RMID, there is another way to directly implement control by adding a flowrate controller (Figure 13). When the flowrate increased, the opening of the valve would be smaller because of the reverse-action of the controller, then the flowrate was reduced, which increased the liquid holdup in the middle vessel. Compared to the CCS with a modified level-set point, the liquid holdup in the middle vessel could be controlled by the flowrate controller and the n-heptane mole fraction could still be as high as 92.0 mol% based on the following analysis and results. Therefore, it was not necessary to specifically investigate the relationship between the level in the middle vessel and n-heptane concentration so that the flowrate-limiting control structure (FLCS) was more applicable. Figure 11c shows that the flowrate of stream TO-MIDV was larger than that of stream RMID in the first 18 h, indicating that the high liquid holdup in the middle vessel was formed during this period. Then, the difference between the flowrate of the two streams decreased and they eventually became almost equal to each other. The fluctuation that occurred at 20 h in the TO-MIDV flowrate curve was caused by the oscillation in the cyclohexane content of the reflux drum. The increase in the cyclohexane content reduced reflux flowrate to the upper section column by the function of CCtop.

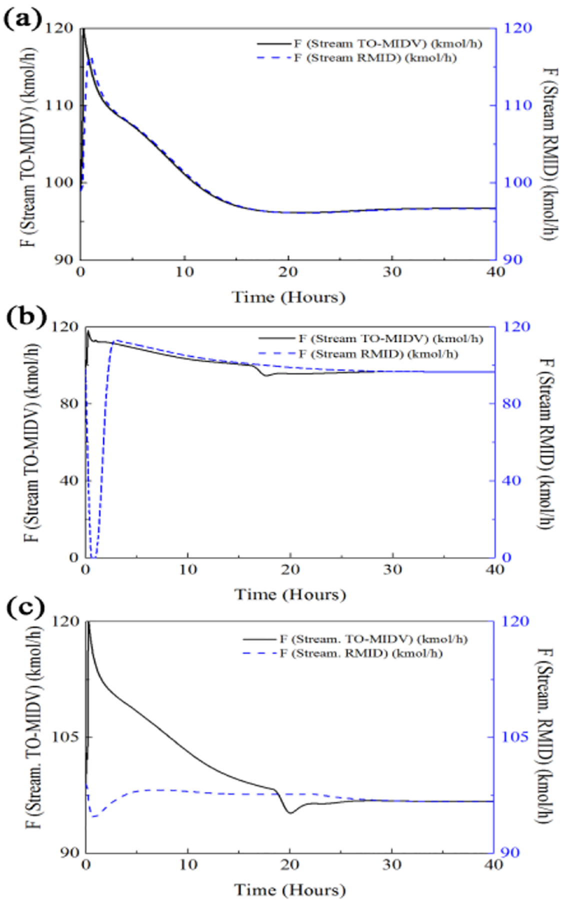

Figure 11. Flowrate comparison of the inlet and the outlet streams of the middle vessel in the composition control structure.

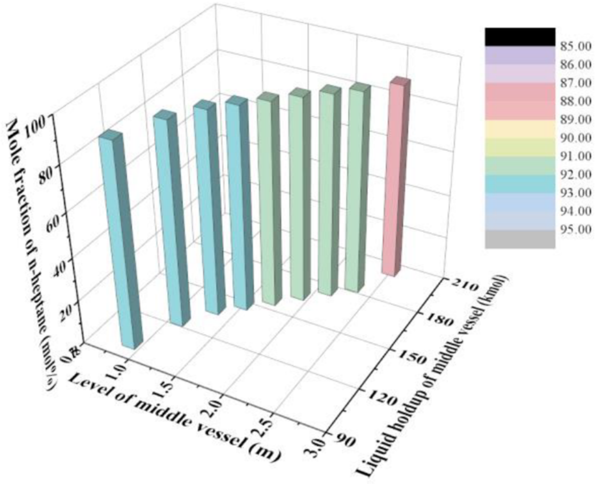

Figure 12. The effects of the level in the middle vessel on n-heptane content and liquid holdup.

Then, the liquid level of the sump in the column dropped, and the opening of valve VUP decreased. Consequently, the flowrate of stream TO-MIDV was reduced by the direct-acting level controller LCbase. In addition, the flowrate-limiting control structure was different from the proposed control structure in our published paper (Zhu *et al*., 2016). The relative volatility of methyl formate/methanol/water is much larger than that of cyclohexane/n-heptane/toluene, which makes the difficulty of separating the latter higher than the difficulty of separating the former. The function of the controllers and the principle of control structures in the two papers are also different.

In the FLCS (Figure 13), the cyclohexane in the reflux drum and the toluene in the sump of the LS column obtained purities of 99.0 mol%, whereas the n-heptane content in the middle vessel was up to 92.0 mol%, which was better than in the previous study (Jain *et al*., 2012). The liquid holdup was 297.5 kmol in the reflux drum, 150.1 kmol in the middle vessel, and 113.9 kmol in the sump. All the results are shown in Figure 14, and they demonstrate that the control structure performed well.

The dynamic control performances of the FLCS were tested by adding initial liquid amount disturbances and initial composition disturbances. The liquid amount disturbances were changed by ±10%. The initial composition disturbances were introduced by changing the initial intermediate component (n-heptane) concentration from 39.4 mol% to 47.3 mol% and 31.5 mol% (±20%). The other two components were split by the original proportion. The composition of cyclohexane was changed from 40.7 mol% to 35.4 mol% and 46.0 mol%, while the composition of toluene was changed from 19.9 mol% to 17.3 mol% and 22.5 mol%. Figure 15a shows the dynamic response of the +10% liquid amount disturbance. The product purities and liquid holdups were controlled stably. In the reflux drum, the liquid holdup was 329.54 kmol, with a composition of 99.0 mol% cyclohexane. The liquid holdup of the middle vessel is 167.79 kmol, with a composition of 92 mol% n-heptane. In the sump of the LS column, the liquid holdup is 128.45 kmol, with 99.3 mol% toluene. The other disturbances are shown in Figures 15b, 16a, and 16b. All the purities of cyclohexane and toluene met the 99.0 mol% mark, and the n-heptane concentration was stable at 92.0 mol%. The controllability of the FLCS performed well based on the dynamic responses.

Table 8. The results of the composition control structure with (1) and without (2) the modified level-set point.

Parameters	CCS^1	CCS^2
X_{cyclo} (mol%)	99.0	99.0
$X_{n\text{-}hep}$ (mol%)	92.0	92.0
X_{tol} (mol%)	99.3	99.3
$M_{reflux\ drum}$ (kmol)	301.5	286.8
$M_{mid\text{-}v}$ (kmol)	147.8	28.5
M_{pot} (kmol)	106.9	123.6

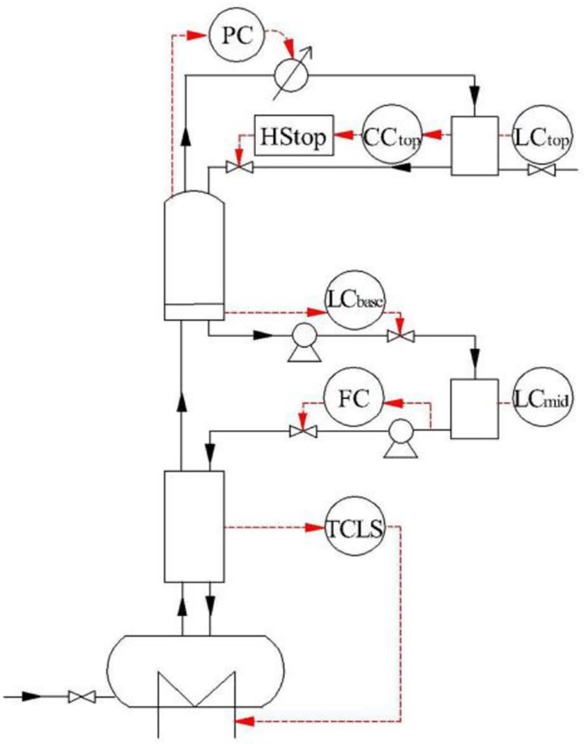

Figure 13. The flowsheet of the flowrate-limiting control structure.

CONCLUSIONS

Conventional batch distillation and MVBD were studied to separate a mixture of cyclohexane/n-heptane/toluene. The modified operations applied in the conventional batch distillation resulted in obtaining a higher purity of product. However, due to the features of the conventional process, the ternary mixture could not be separated simultaneously, which would keep the batch time long.

MVBD, a more flexible batch process, allowed the collection of the intermediate component in the middle vessel and made it possible to separate the ternary system at the same time. The CCS with a modified level-set point and the FLCS were explored with the goal of achieving high product purity and liquid holdup. Based on the simulation results, the product purities achieved through using the two control structures were the same, although the liquid holdup was slightly different. The liquid holdup of the middle vessel and the sump in the FLCS were higher than in the CCS with a modified level-set point. In addition, when using the FLCS to control the process, it was not necessary to specifically investigate the relationship between the liquid level in the middle vessel and n-heptane concentration, which made FLCS more applicable than CCS with a modified level-set point. Therefore, when we use the MVBD to separate ternary mixtures with low relative volatility, similar to the ternary system in this paper, FLCS can be preferentially chosen. To assess the controllability of the control structure, liquid amount disturbances and initial composition disturbances were introduced. Based on the dynamic responses, all the purities and liquid holdups of the products were stable at a high level, which showed good performance of the control structure. The FLCS could potentially be carried out in practice, and it offers guidelines for practical processes. Both the level controllers and the flowrate controllers are already used in industry. For the composition controller, although it is difficult to achieve accurate online detection, the purity of the light component in the control structure was kept sufficiently high during the whole process based on the simulation results. Therefore, some other methods, such as gas chromatography, could be used, and the time delay caused by determining the purity would not impact the benefits of the process.

ACKNOWLEDGEMENTS

Support from the projects of the National Natural Science Foundation of China (Project 21676152 and Project 21306093) are gratefully acknowledged.

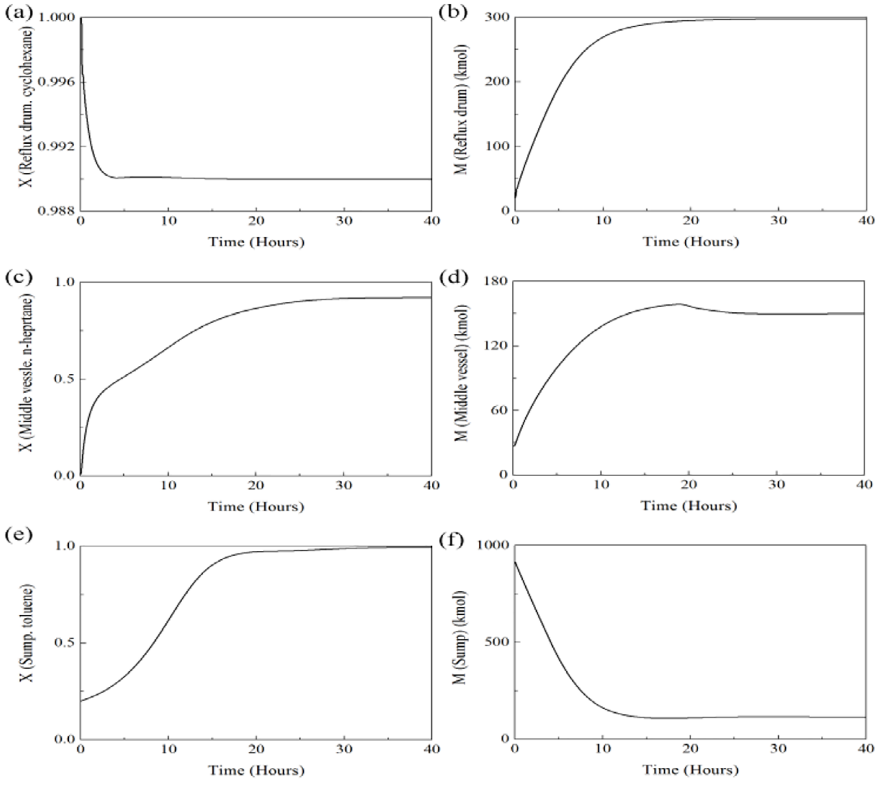

Figure 14. Results of the flowrate-limiting control structure.

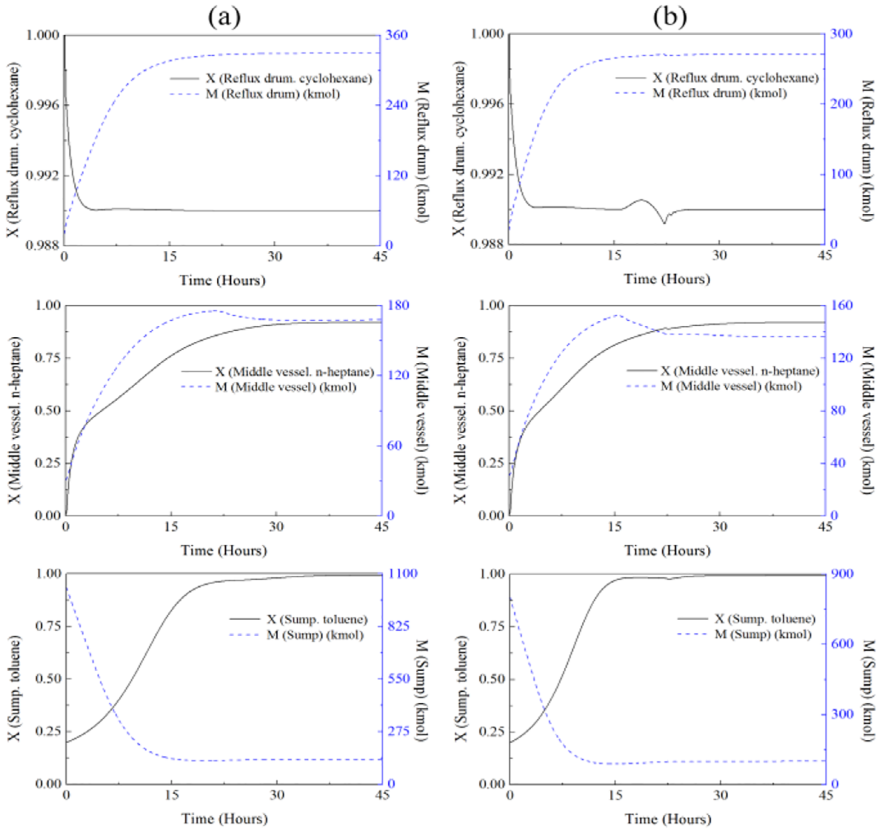

Figure 15. Dynamic responses of the liquid amount disturbances: (a) +10%; (b) -10%.

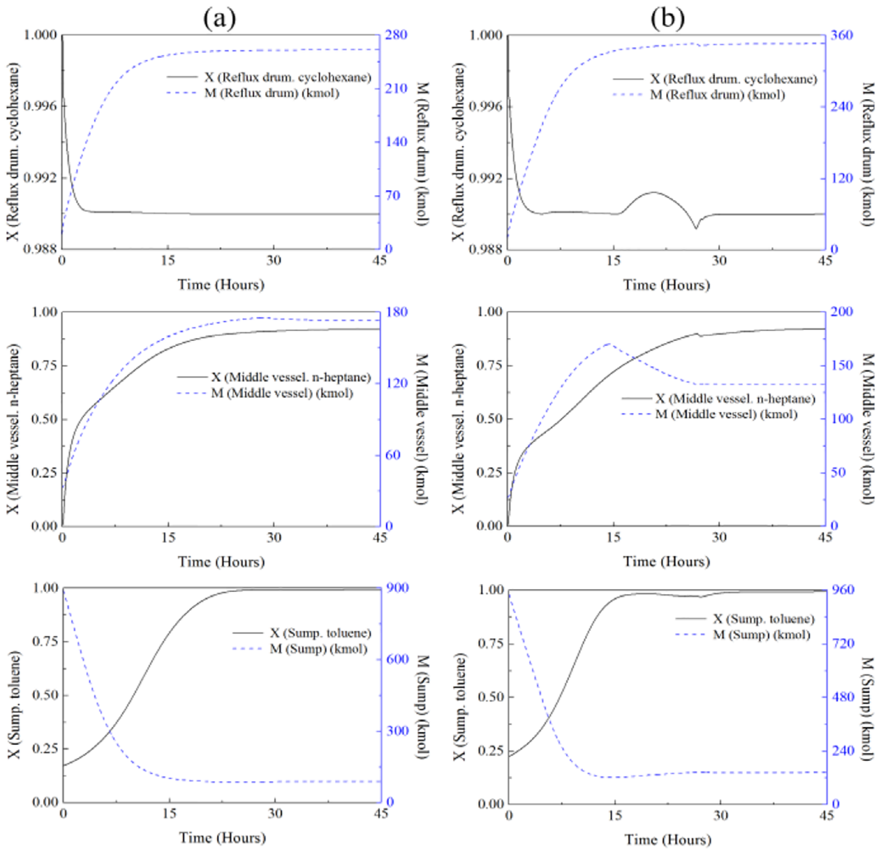

Figure 16. Dynamic responses of the initial composition disturbances: (a) +20%; (b) -20%.

NOMENCLATURE

CCS - Composition control structure
F - Mole flowrate (kmol/h)
F_{cyclo} - Cyclohexane mole flowrate (kmol/h)
F_{d} - Distillate mole flowrate (kmol/h)
$F_{n\text{-}hep}$ - n-heptane mole flowrate (kmol/h)
F_{tol} - Toluene mole flowrate (kmol/h)
F_{total} - Total mole flowrate (kmol/h)
FLCS - Flowrate-limiting control structure
LCS - Level control structure
LS - Lower section
M - Liquid holdup (kmol)
$M_{mid\text{-}v}$ - Liquid holdup of the middle vessel (kmol)
M_{pot} - Liquid holdup of the pot (kmol)
$M_{reflux\ drum}$ - Liquid holdup of the reflux drum (kmol)
MVBD - Middle-vessel batch distillation
T - Temperature (ºC)
T_{hm} - Temperature of heat medium (ºC)
TCS - Temperature control structure
US - Upper section
X - Mole fraction (mol%)
X_{cyclo} - Cyclohexane mole fraction (mol%)
$X_{n\text{-}hep}$ - n-Heptane mole fraction (mol%)
X_{tol} - Toluene mole fraction (mol%)

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