

Design of a Fully Heat-Integrated Pressure-Swing Distillation for Close-Boiling Separation

Zhijun Xi, Chengtian Cui, Jincal Chen, Siyao Liu, Jinsheng Sun*

School of Chemical Engineering and Technology, Tianjin University, Tianjin, China, 300072
 jssun2006@vip.163.com

Enlightened by recent progress in pressure-swing distillation (PSD) and double-effect heat integration, this work combines these two concepts for close-boiling separation, by using two fully heat-integrated columns operating at different pressure plus a recycle stream. The developed fully heat-integrated pressure swing distillation (FHIPSD) is therefore proposed, followed by two close-boiling binary systems with different relative volatility and latent enthalpy as case studies. The main advantage of the FHIPSD is to lower the separation difficulty of each column, circumventing manufacturing a very high column. For certain feed conditions, the FHIPSD can save energy cost and provide higher-temperature residual heat for further heat integration, compared to a conventional distillation column (CDiC).

1. Introduction

Distillation is a main unit operation for the separation and purification of fine chemicals, widely used in petrochemical industry (Cui and Sun, 2017). As an energy-intensive operation, distillation generates more than 50% of plant operating cost (Cui et al., 2017a). Besides, it possesses the largest scale equipment on-site and thus cost-intensive as well (Cui et al., 2017b). With the increasing global industrial growth, distillation has to be improved to be more cost saving (Cui et al., 2016).

Close-boiling mixtures are commonly encountered in the fine-chemical and specialty industries (Long et al., 2013). Generally, the x - y diagram helps analyzing the difficulty of separation (Seider et al., 2017). For a close-boiling system, the much more limited deviation between the equilibrium and the diagonal lines means an extreme separation difficulty in a conventional distillation column (CDiC) (Liu et al., 2017). Normally, this situation calls for a great number of stages and considerable energy consumption. In order to lower separation cost, extractive distillation (ED) is usually used to enhance the relative volatility of the components with an additional solvent, but occasionally the added solvent can cost much more energy consumption. On the other hand, pressure-swing distillation (PSD) is commonly used to separate pressure-sensitive azeotropes (Tao et al., 2017). This method does not involve any solvents and thus preferred than ED in some azeotropic separation cases. However, PSD is normally regarded as a specific method for azeotropic separation rather than close-boiling separation. This study tries to introduce the PSD concept for close-boiling separation. As a PSD configuration has two columns, it is naturally prone to double-effect heat integration (Cui et al., 2017c). In this study, a fully heat-integrated pressure swing distillation (FHIPSD) process is developed for close-boiling separation. This process is also known as the distillation with recycle process (DRP) (Cui et al., 2017d), and has been previously applied in organosilicon monomer distillation (Sun et al., 2012). Although FHIPSD has been drew less attentions in academics, it is very useful in engineering practices. From two case studies, it is shown that the FHIPSD can significantly lower the required column stage. However, the additional recycle stream in FHIPSD introduces unexpected extra energy consumption, thus it is necessary to analyze this method according to the actual situation before putting it into use. This study tests the performance of FHIPSD with two case studies – methylcyclopentane (MCP) /cyclohexane (CH) and isobutanol /*n*-butanol. For certain feed conditions, it is observed that FHIPSD could also save part of energy cost over a CDiC.

2. Fully heat-integrated pressure-swing distillation

The basic concept of PSD is to combine a high-pressure column (HPC) and a low-pressure column (LPC) with a recycle stream, which can reduce stage requirement because the bottoms and the distillate of each column do not require stringent purity specifications simultaneously. Besides, the HPC overhead vapor can be used to heat LPC reboiler in a fully heat-integrated manner. At the same time, the product composition of the recycle stream should be consistent with the composition on the feed stage to avoid back-mixing effect. In general, the FHIPSD can be divided into the light split forward (LSF) and the heavy split forward (HSF) depending on the splitted component. When applying this method, it is necessary to make a reasonable adjustment of operating pressures on two columns and the circulation flow rate for minimum energy consumption.

The flowsheet of FHIPSD-LSF and FHIPSD-HSF is shown in Figure 1. For a close-boiling system A/B (A and B are light and heavy key component, respectively), the crude feed enters the first column after mixing with the recycle stream, reaching the separation target of A in distillate. Because the residue of the first column is designed to contain a certain amount of A, it lowers the operating temperature of the column and, more importantly, the separation difficulty as well, which is particularly benefit for thermal unstable components that decompose or polymerize at elevated reboiler temperatures. When the residue is separated in a subsequent column, the distillate is designed to be the same A/B composition as the feed of the first column, circulating back to the first column feedstock. Pure B yields within residue with a reduced separation difficulty as well. The HPC condenser and LPC reboiler are combined into one process-to-process heat exchanger to save energy. This flowsheet is suitable for the situation where the light component flow rate is relatively high. Likewise, FHIPSD-HSF is similar to its LSF counterpart. The only different lies in its distillation sequence.

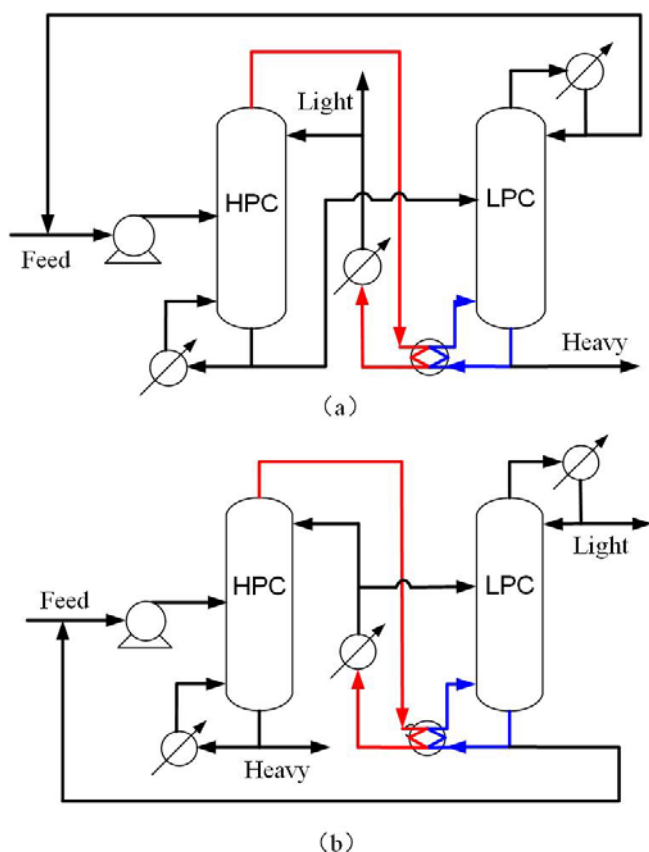


Figure 1: (a) FHIPSD-LSF and (b) FHIPSD-HSF

3. Case studies

In order to test the performance of the FHIPSD method, Aspen Plus was applied to simulate the process of CDiC, FHIPSD-LSF and FHIPSD-HSF for specified close-boiling system, respectively. In-built RadFrac model was selected for simulating distillation columns. MCP/CH and isobutanol/n-butanol with different feed

compositions are selected as two case studies. Feed molar flow rate is set as 100 kmol/h, and the NRTL thermodynamic model is selected to calculate the vapor-liquid equilibrium (VLE).

3.1 Methylcyclopentane/Cyclohexane

For this case, although vacuum operation appears to be reasonable in the column, it is generally not justified from the standpoint of process economics because it poses strict requirements on the cold utility and the construction of the vacuum column. Therefore, the operating pressures of the CDiC and the FHIPSD LPCs are set at 110 kPa. And the operating pressures of the FHIPSD HPCs are postulated to be 240 kPa. Figure 2 presents the x-y diagram of the MCP/CH system at 110 kPa and 240 kPa, respectively. This diagram demonstrates this system is quite pressure-insensitive. Three feed compositions – 0.95/0.05, 0.5/0.5 and 0.05/0.95 of MCP/CH – are tested by CDiC, FHIPSD-LSF and FHIPSD-HSF, respectively. The corresponding separation targets are 99.9 mol% for MCP and CH.

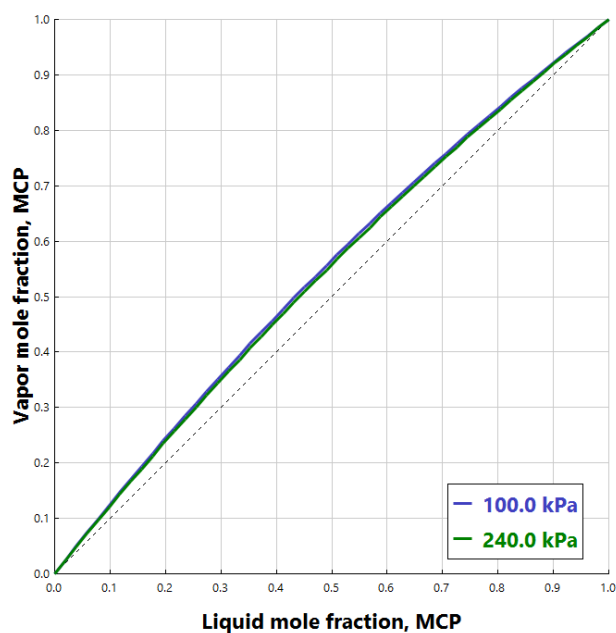


Figure 2: x-y diagram of the MCP/CH system at different pressures

Table 1: Summarized results of MCP/CH system simulation in the 0.95/0.05 feed composition

	CDiC	LSF-HPC	LSF-LPC	HSF-HPC	HSF-LPC
Number of stages	130	90	55	85	100
Feed stage	90	79	14	41	49
Overhead pressure, kPa	110.0	240.0	110.0	240.0	110.0
Overhead temperature, °C	74.49	102.46	74.89	102.59	74.49
Bottom pressure, kPa	122.5	247.9	113.8	247.9	113.8
Bottom temperature, °C	87.03	105.13	84.50	113.52	79.90
Condenser duty, kW	3480	2640	1145	390	3246
Reboiler duty, kW	3480	3923	0	3637	0
Reflux ratio	3.50	4.15	2.54	2.35	3.24
Flow rate of recycle stream, kmol/h		40.0		40.0	

When the feed composition is 0.95/0.05, the summarized results are shown in Table 1. For this feed condition, the LSF shows better performance: Although more energy is required, the number of theoretical stage of each column is reduced, thereby reducing the column height. Simultaneously, a large amount of residual heat (~102 °C) is generated, which can be used to drive other devices.

Table 2: Summarized results of MCP/CH system simulation in the 0.50/0.50 feed composition

	CDiC	LSF-HPC	LSF-LPC	HSF-HPC	HSF-LPC
Number of stages	125	90	65	85	65
Feed stage	62	79	6	16	56
Overhead pressure, kPa	110.0	240.0	110.0	240.0	110.0
Overhead temperature, °C	74.49	102.46	78.77	105.22	74.49
Bottom pressure, kPa	122.5	247.9	113.8	247.9	113.8
Bottom temperature, °C	87.21	110.53	84.68	113.71	79.90
Condenser duty, kW	3347	746	2812	789	2699
Reboiler duty, kW	3357	3633	0	3562	0
Reflux ratio	7.21	7.86	3.65	2.48	5.69
Flow rate of recycle stream, kmol/h		73.7		73.6	

For the 0.50/0.50 case, the calculation results are shown in Table 2. Three processes have similar reboiler duty, but the FHIPSD can produce certain amount of higher temperature residual heat. In addition, the FHIPSD requires two columns with lower stage number, thus making it practical in engineering construction. However, it is still necessary to comprehensively evaluate these processes in economic criterion.

Table 3: Summarized results of MCP/CH system simulation in the 0.05/0.95 feed composition

	CDiC	HSF-HPC	HSF-LPC
Number of stages	140	95	65
Feed stage	41	14	49
Overhead pressure, kPa	110.0	240.0	110.0
Overhead temperature, °C	74.64	110.75	74.64
Bottom pressure, kPa	122.5	247.9	113.8
Bottom temperature, °C	87.21	113.71	84.19
Condenser duty, kW	3042	2238	1035
Reboiler duty, kW	3060	3417	0
Reflux ratio	73.59	8.14	24.65
Flow rate of recycle stream, kmol/h		40.0	

When the feed composition is 0.05/0.95, LSF requires a very large amount of recycle stream to meet the separation requirements, which is engineering infeasible. Therefore, only CDiC and HSF processes are considered. The simulation results are shown in Table 3. It can be seen that the effect of HSF is similar to the LSF in Table 1. Compared to the CDiC, the column height in HSF is reduced in the cost of slightly more energy input, but much more medium-temperature residual heat can be provided for use.

3.2 Isobutanol/n-butanol

In this case, the operating pressure of the HPCs is 270 kPa, whereas the CDiC and LPCs are still operated at 110 kPa. The x-y diagram of the isobutanol/n-butanol system at 110 kPa and 270 kPa is indicated in Figure 3. It can be demonstrated from the x-y diagram that this system is quite pressure-sensitive. When the molar fraction of light component is low, the curve approaches the diagonal line. Therefore, the FHIPSD approach is not suitable for the mixture with high content of heavy component. Hence, the feed composition discussed is 0.95/0.05 and 0.5/0.5 of isobutanol/n-butanol with each component purified to 99.9 mol%.

When the feed composition is 0.95/0.05, the simulation results are listed in Table 4. In this case, LSF has excellent performance: compared to the CDiC, not only the theoretical stages requirement of each column are reduced, but also the energy consumption, which is reduced by ~25%. In addition, more medium-temperature residual heat (~137 °C) is generated. On the other hand, HSF process does not perform well in this situation. When the feed composition is 0.50/0.50, the simulation results are shown in Table 5. Due to the influence of pressure on VLE, LSF performs better than other processes. Compared to the CDiC, LSF can save ~20% of energy consumption, but HSF does not have a good performance for this case.

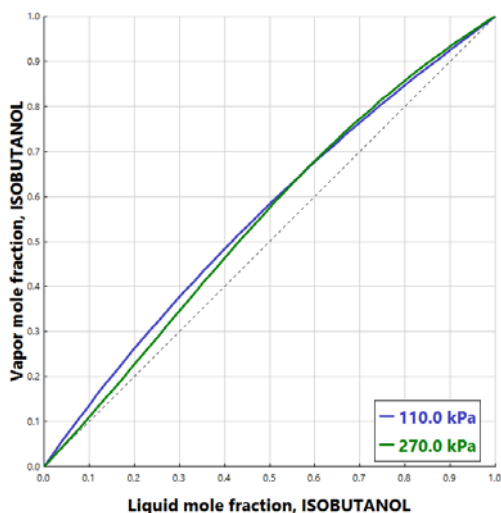


Figure 3: x - y diagram of the isobutanol/ n -butanol system at different pressures

Table 4: Summarized results of isobutanol/ n -butanol system simulation in the 0.95/0.05 feed composition

	CDiC	LSF-HPC	LSF-LPC	HSF-HPC	HSF-LPC
Number of stages	100	60	50	60	90
Feed stage	70	54	12	6	39
Overhead pressure, kPa	110.0	270.0	110.0	270.0	110.0
Overhead temperature, °C	109.94	137.42	110.34	137.64	109.94
Bottom pressure, kPa	119.4	275.8	113.8	275.8	113.8
Bottom temperature, °C	122.46	140.25	120.83	149.19	111.29
Condenser duty, kW	4050	1638	1313	0	3981
Reboiler duty, kW	4042	3130	0	3254	720
Reflux ratio	2.68	1.85	1.86	0.96	2.64
Flow rate of recycle stream, kmol/h		40.0		50.0	

Table 5: Summarized results of isobutanol/ n -butanol system simulation in the 0.50/0.50 feed composition

	CDiC	LSF-HPC	LSF-LPC	HSF-HPC	HSF-LPC
Number of stages	75	60	55	240	60
Feed stage	33	50	8	5	51
Overhead pressure, kPa	110.0	270.0	110.0	270.0	110.0
Overhead temperature, °C	109.94	137.42	114.42	140.85	109.94
Bottom pressure, kPa	119.4	275.8	113.8	275.8	113.8
Bottom temperature, °C	122.46	147.24	121.06	149.23	115.43
Condenser duty, kW	4509	752	2865	8158	2834
Reboiler duty, kW	4461	3662	0	11020	0
Reflux ratio	6.79	5.17	1.67	9.78	3.92
Flow rate of recycle stream, kmol/h		96.0		49.0	

3.3 Case Summary

Through the above two cases, it can be observed that the FHIPSD is capable of reducing stage requirement of each column, thereby lowering column height, and simultaneously, providing higher temperature residual heat that can be used in heat integration with other devices. On the other hand, it is demonstrated for the pressure-sensitive isobutanol/ n -butanol system, FHIPSD can provide good energy-saving performance. But it is still uncertain if this rule can be held for other pressure-sensitive systems. For pressure-insensitive systems, the implementation of FHIPSD can lead to an increase in energy consumption and total stage requirement. Therefore, the use of the FHIPSD method requires serious evaluation. Also, it is necessary to determine which

separation sequence (LSF or HSF) is better for use, based on the feed composition and its corresponding VLE relationship.

4. Conclusions

This work introduces an unobtrusive method for close-boiling separation – the FHIPSD method, which has high value in practical industrial design because it can reduce the column height. CDIC, FHIPSD-LSF and FHIPSD-HSF are compared by two cases. It is observed that the optimal choice has a close relationship with the VLE trend and the feed composition. For pressure-sensitive system, the FHIPSD might have energy-saving effect. Another advantage of this approach is export higher temperature residual heat that can be used in further heat integration.

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