

## Experimental evaluation of a modified fully thermally coupled distillation column

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**Abstract**—A modified fully thermally coupled distillation column replacing the conventional distillation system is introduced, and its performance is experimentally evaluated for the stable operation of the column. The existing distillation system is modified to an energy-efficient distillation column for the reduction of investment cost and energy requirement. The experiment is conducted for the separation of methanol, ethanol and *n*-propanol mixture using a 4-in sieve tray column. The temperatures at seven different locations of the column are measured to monitor the column operation, and the measurements indicate that the column is stably operable. The stable operation proves that neither compressor nor pump is necessary for the vapor or liquid flow between tray sections. The experimental result is compared with that of the HYSYS simulation to show how satisfactory the separation is. The modeling of temperature variation provides a successful prediction of the temperature variation.

Key words: Thermally Coupled Distillation, Energy-efficient Distillation, Multi-component Distillation, Experimental Evaluation

### INTRODUCTION

The divided wall column (DWC), a kind of fully thermally coupled distillation column (FTCDC), has been widely employed in many processes, and its energy efficiency and operability have been proved in commercial applications [1]. Though the energy efficiency and operability of the DWC have been confirmed from the practical applications, the replacement of the current distillation column with the DWC is not simple due to the construction of the new column. By utilizing the existing columns of the conventional system to the FTCDC, a modified structure has been proposed and applied to the BTX process [2].

In the design of the FTCDC, the minimum reflux flow rates for a main column and prefractionator have been calculated in many studies [3-5]. Because the FTCDC has interlinking streams between the main column and prefractionator, the conventional design procedure for distillation column cannot be used in the FTCDC design. The three-column model was introduced for the FTCDC design by Triantafyllou and Smith [6], and was improved by including semi-rigorous material balance and equilibrium relation [7]. The main procedure in the design is the division of the main column into two separate columns to eliminate the interlinking streams.

On the other hand, the tray composition profile of equilibrium distillation was utilized in the structural design of the FTCDC to determine the interlinking location and feed and side draw trays [8]. A liquid composition profile following the equilibrium distillation line gives the minimum tray structure having ideal thermodynamic efficiency. If the assumption of the ideal tray efficiency and total reflux operation is applied to the distillation column, the col-

umn requires the minimum number of trays for a given separation of feed mixture. When the distillation line of the FTCDC matches the profile, the highest efficiency is available by eliminating the feed tray mixing and the remixing of intermediate component, which are the sources of the efficiency reduction [6]. The structural design using the minimum tray has been applied to various distillation systems of ternary [8-13] and quaternary mixtures [10,14,15]. To improve the control of the FTCDC, the unidirectional flow scheme [16] and the profile position control [17] have been introduced recently.

In this study a modified FTCDC utilizing the conventional distillation columns is explained, and its design procedure is addressed. The designed system is applied to a pilot-scale distillation column having 4-inch sieve trays for the experimental evaluation of the column. The operability and separation performance are experimentally investigated, and the results are compared with those of the HYSYS simulation. A ternary mixture of methanol, ethanol and *n*-propanol is employed in the experiment. Also, a prediction model for the temperature measurements is presented, and its performance is analyzed.

### COLUMN DESCRIPTION

In a conventional distillation system with direct sequence for ternary separation, the lightest component in the feed is produced as the overhead product of the first column. The mixture obtained in the bottom having the intermediate and heaviest components is fed to the second column to be separated into the other two products. When all the three products are produced from a single column, the mixture of the intermediate and heaviest components is not vaporized twice unlike the conventional system reducing energy requirement. In fact, the divided wall column (DWC) yielding the three

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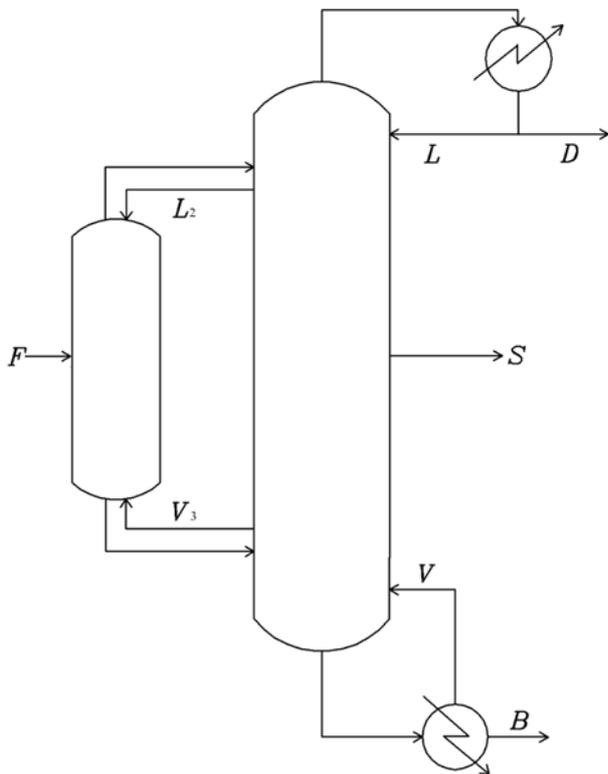


Fig. 1. A schematic diagram of a fully thermally coupled distillation (Petlyuk) column for ternary separation.

products in the single column consumes about 30% less energy than the conventional system. Because the feed composition is quite different from that of the side product, a separate section of trays is necessary avoiding the mixing of the two different streams of feed and side draw. The section handling the feed is called a pre-fractionator, but the whole system operates like a single column because there are one reboiler and one condenser in the system.

The Petlyuk column and the DWC have the same principle of ternary separation known as the fully thermally coupled distillation column (FTCDC) described in Fig. 1. The manipulation of the inter-linking vapor and liquid flows requires pressure balancing between the pre-fractionator and main column difficult to handle. Due to the successful operation of the DWC at the practical plants, the field engineers prefer the DWC to the Petlyuk column for their new projects. However, the utilization of the existing distillation columns is not possible in the DWC application, because the middle section with divided wall has quite different structure from the conventional column.

Instead of the main column of the FTCDC, three separated columns can be used as in the modified FTCDC illustrated in Fig. 2. In this configuration either tray or packed column can be utilized, and therefore the two binary columns of the conventional distillation system are placed as the columns, C1 and C2, in the figure. Because the modified FTCDC replaces the two existing binary columns, their utilization reduces the investment cost required for the new system building. The upper and lower sections of the main column, denoted E1 and E2 in Fig. 2, have to handle the combined flow of vapor in the two binary columns of the conventional system,

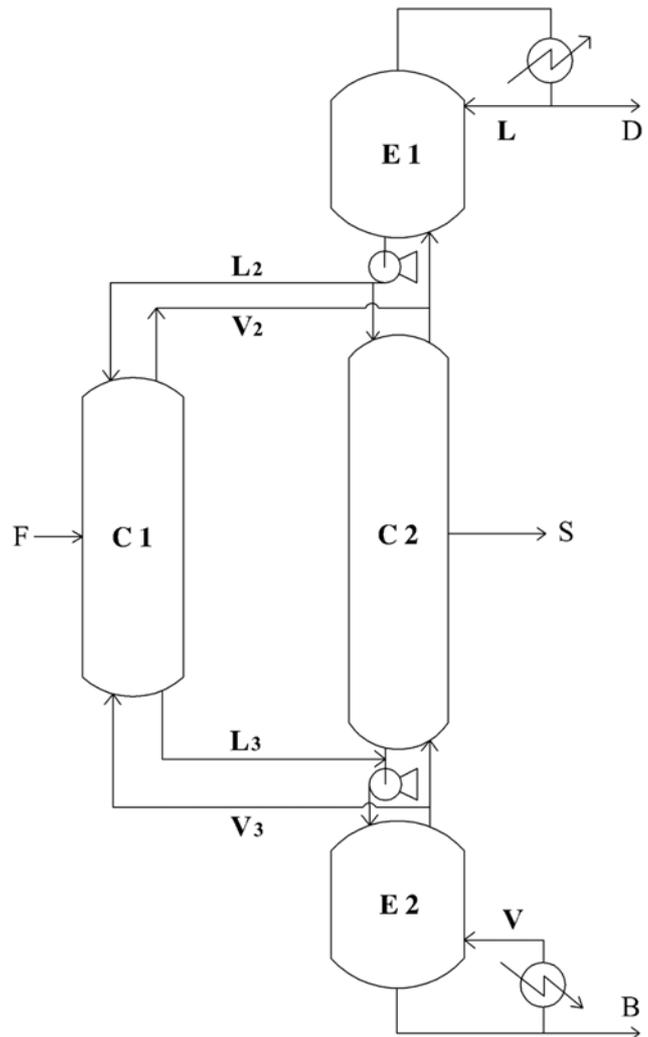


Fig. 2. A schematic diagram of a modified fully thermally coupled distillation column for ternary separation.

and therefore larger diameter columns are necessary. These columns having small tray numbers are required to be newly built. By setting the pressure of the column E2 the highest and that of E1 the lowest, no compressor is necessary for the vapor flow between the columns. Instead, two pumps much simpler than a compressor in terms of cost and operation are used for the liquid flow. Both the liquid and vapor splits between the pre-fractionator and main column are adjusted in the modified distillation system. This expands the operating range of the modified distillation system. Though a large change of feed condition is not handled, minor change can be easily handled with the manipulation of the splits, which is not available with the DWC.

## DISTILLATION COLUMN DESIGN

The design of the modified distillation system for the ternary mixture of methanol, ethanol and *n*-propanol begins with the conventional system design using the standard procedure. The conventional distillation columns are used as the pre-fractionator and middle main column. When the commercial design software HYSYS is

**Table 1. Column structure and operating conditions. Tray numbers are counted from top**

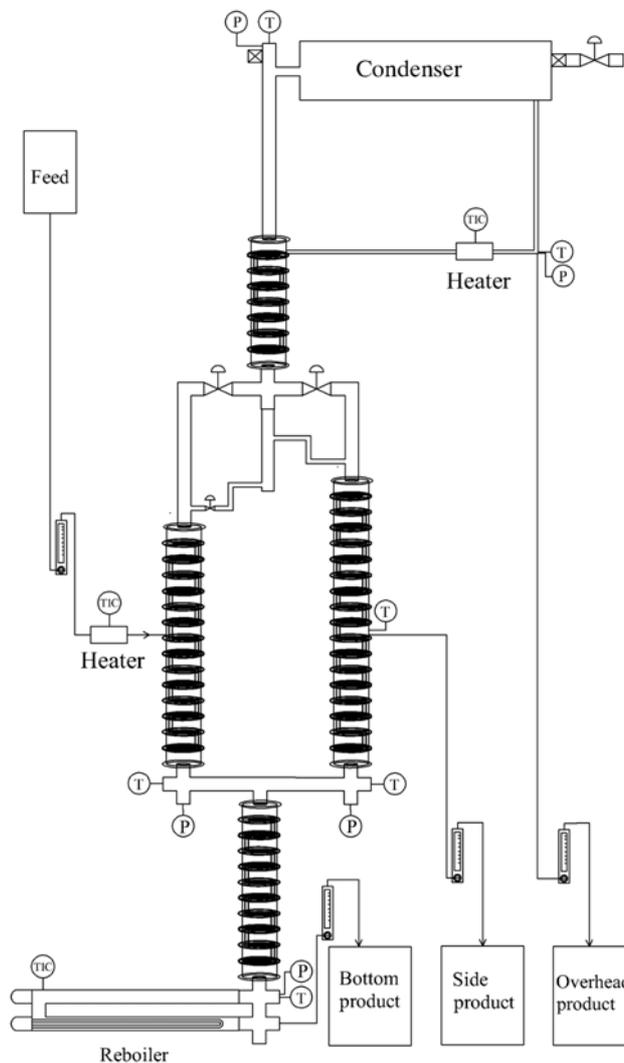
Name	Experimental column			
	Prefract.	Up_main	Mid_main	Low_main
<b>Structural</b>				
Number of tray	14	6	16	10
Feed/side product	8		10	
<b>Operating</b>				
Feed (mL/min)	30			
Overhead (mL/min)		11		
Bottom (mL/min)				7
Side (mL/min)			11	
Reflux (mL/min)	11		48	
Heat duty (kW)		0.61		2.02

used, the numbers of trays and column pressure are necessary for the column design. The condenser pressure was set at the atmospheric pressure, because the boiling point of methanol is about 40 degrees Celsius above the room temperature-the temperature of readily available cooling water. In this experiment the number of trays was limited due to the height of the laboratory, and therefore the total number was determined accordingly. Table 1 lists the numbers of trays. The operational variables for the given product specification were computed from the HYSYS simulation.

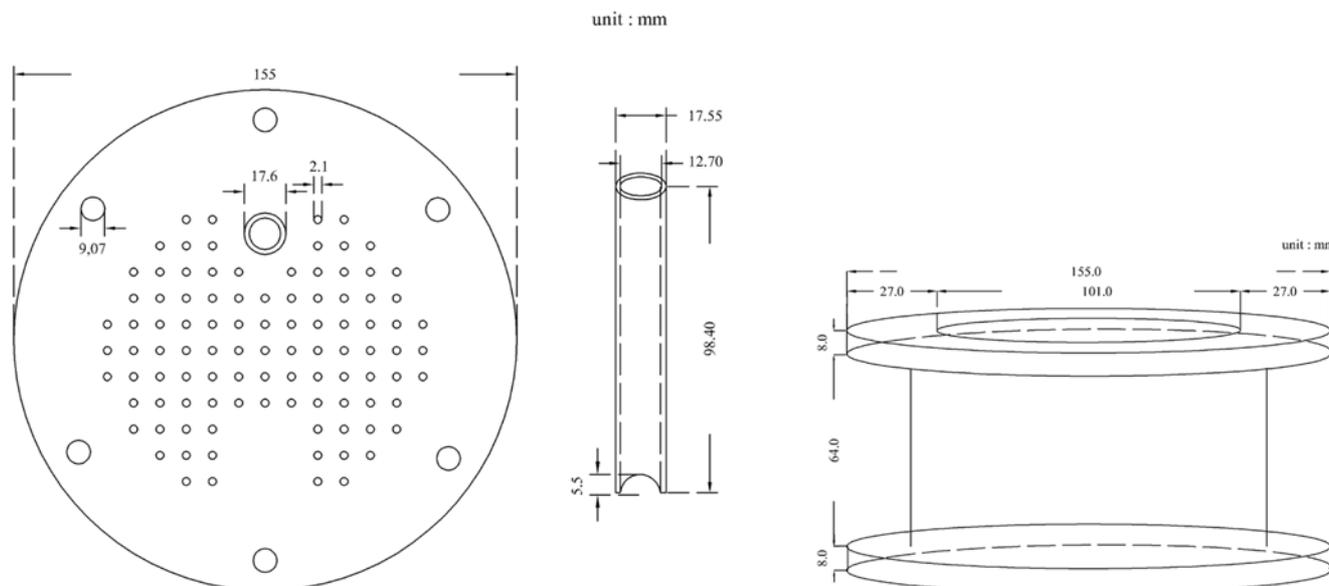
**EXPERIMENTAL**

**1. Column Construction**

The experimental column was composed of four sections of distillation trays of 4 in sieve trays shown in Fig. 3. For the convenience of experimental operation no pump was used, and the liquid was transported by its gravitation. The layout of the experimental equipment is demonstrated in Fig. 4. Four sections of distillation



**Fig. 4. A schematic diagram of experimental distillation column.**



**Fig. 3. A schematic diagram of sieve tray used in the experiment.**

columns were placed in the middle as denoted C1, C2, E1 and E2 in Fig. 2. Two large valves were installed between the upper main column and middle main column and prefractionator for the flow control of vapor, and one small valve for the control of liquid flow to the prefractionator. The flow rates of feed and three products were adjusted and measured with four rotameters a valve attached. A set of electric heaters was installed at the reboiler. Temperature controllers were used for the steady supply of heat. A glass tube was attached at the reboiler for the level measurement. The feed and reflux were also heated using separate electric heaters with temperature controllers. Temperatures of three products and bottom temperatures of the prefractionator and middle main column were measured, and the measurements were collected using a PC. The pressure measurements were used to detect the liquid loading at the trays and to monitor the vapor flow.

## 2. Experimental Procedure

The feed tank was filled with a mixture of methanol, ethanol and *n*-propanol. The feed was supplied until the heaters at the reboiler were fully immersed with the liquid. The electric heat was supplied to the reboiler while its temperature was measured. When the liquid level began to lower, the feed was supplied at the given rate. After the temperature of side draw tray reached its operating temperature, the products of side and bottom were continuously drawn at the given rates. Note that the methanol and ethanol contained in the feed have to be removed for the bottom product of high concentration of *n*-propanol. When the top tray temperature reached its operating temperature, the overhead product was drawn. The measured temperatures were stored in the PC, and retrieved for the data analysis later. The composition of feed and three products was measured with a gas chromatograph (Agilent Model 5892A, U.S.A.).

## RESULTS AND DISCUSSION

Though the composition of three products is their specification, the on-line measurement of the composition is practically difficult. Instead, tray temperature is widely used for the monitoring the operating condition of a distillation column in the industrial applications.

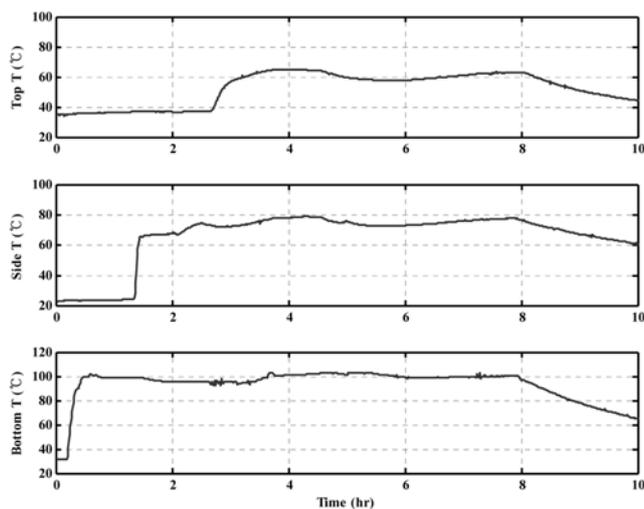


Fig. 5. Temperature variations of top and side draw trays and reboiler.

The variation of product composition is predicted from the temperature change at the tray of product draw. The temperatures of top tray, side draw tray and reboiler were measured to monitor the operation of the modified FTCDC in this study (Fig. 5). The top figure represents the variation of top tray temperature, the middle does that of the side draw tray, and the bottom for the reboiler. The experiment was conducted for 10 hours. The temperature of reboiler increased first, and that of side draw tray increased about an hour later. Finally, the top tray temperature was raised an hour-and-a-half later from the side temperature increase. After the temperature was raised to its steady state value, it was maintained at the level for eight hours from the beginning of the experiment. Then the heat supply to the reboiler was cut to reduce the tray temperatures. The composition of feed and three products was measured during the steady state operation. While the temperature at the reboiler was stable, the top tray temperature had dropped a little in the middle of the steady state operation. The drop was also observed in the side draw tray. It is presumed to come from an increase of reflux flow when the liquid holdup in the condenser reached to its limit. Note that no reflux drum was installed in the experimental setup.

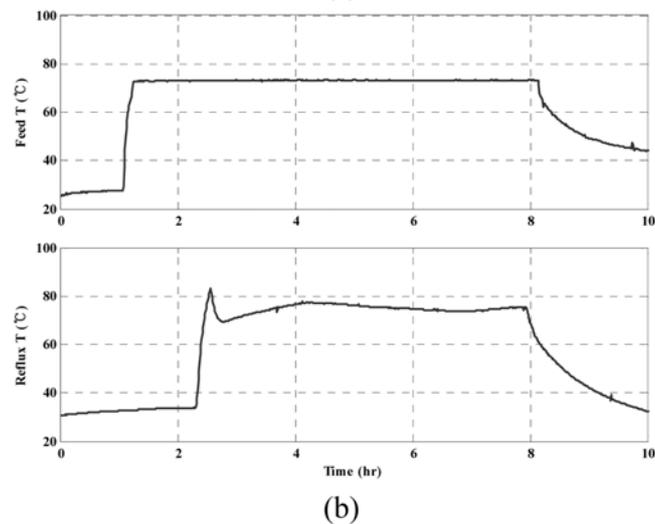
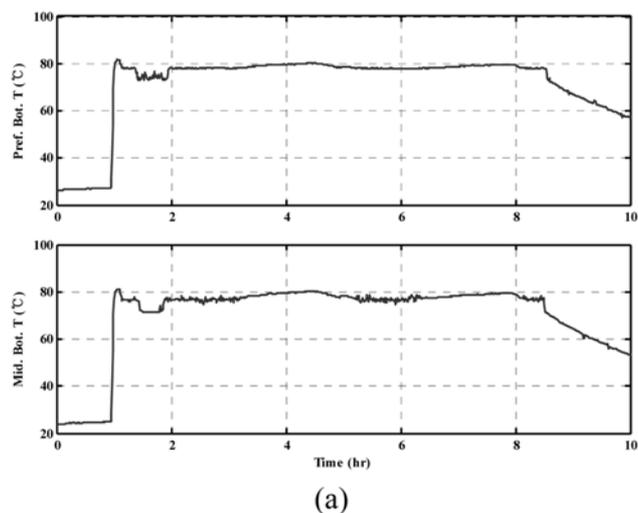


Fig. 6. Temperature variations of bottom trays of prefractionator and middle main column (a), and feed and reflux (b).

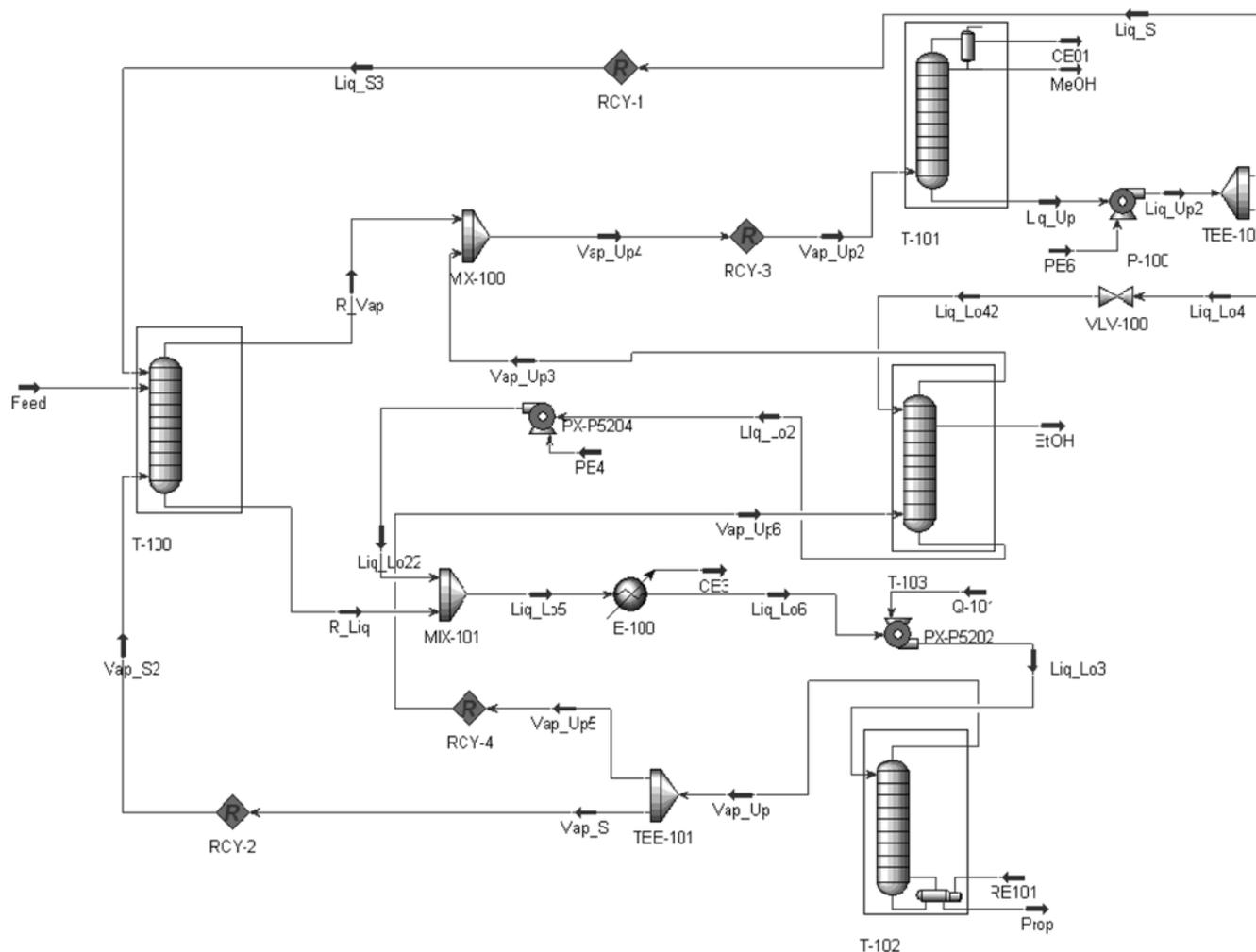


Fig. 7. A process flow diagram of the modified FTCCD for simulation.

The temperatures of bottom trays of the prefractionator and middle main column are shown in Fig. 6(a). These two temperatures are used for the distribution control of vapor flow from the lower main column to the prefractionator and middle main column by manipulating the vapor flow valves. The temperature variation indicates that the split of the vapor flow was stable at steady state operation. The stable temperature after the trays were heated demonstrated that the column operation was stable. For the reference the temperatures of feed and reflux are shown in Fig. 6(b). The temperatures of feed and reflux were adjusted with separate electric heaters, and the measurements illustrated that the temperature control was satisfactory.

The operating condition of the experimental distillation column is listed in Table 1. The flow rates of feed and products were manipulated by manual operation, and the heat supply to the reboiler was adjusted with an electric transformer. The difference between the reboiler heat supply and condenser duty is presumed to be heat loss around the tray sections. Though a glass wool insulation of 5 cm in thickness was installed in the whole column, a large amount of heat was lost. To examine the experimental result of product specification, the HYSYS simulation was conducted by using the experimental tray structure and operating condition listed in Table 1. The

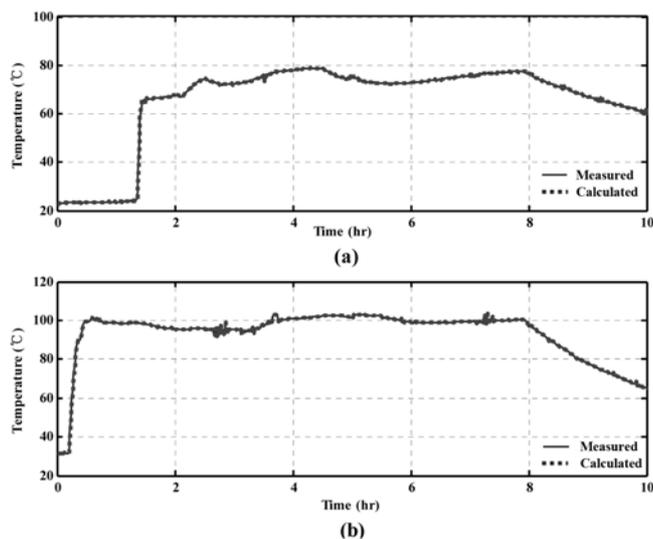
Table 2. Specifications of feed and products from experimental and simulation. Unit is in mole fraction

Components	Feed	Overhead	Side	Bottom
Experimental				
Methanol	0.346	0.711	0.167	0.004
Ethanol	0.409	0.286	0.779	0.128
<i>n</i> -Propanol	0.245	0.003	0.054	0.868
Simulation				
Methanol	0.346	0.839	0.080	0.000
Ethanol	0.409	0.161	0.898	0.061
<i>n</i> -Propanol	0.245	0.000	0.022	0.939

tray efficiency of 70% was considered in the simulation. Fig. 7 shows the process flow diagram used in the computation. The experimental results measured with the gas chromatograph and the computed values are summarized in Table 2. With the same feed composition and operating condition applied, the compositions of intermediate component in side draw from the experiment and simulation are close. When the composition of the intermediate component in feed is compared with the experimental result, it is found that the separa-

**Table 3. Model parameters**

Temperature	$a_1$	$a_2$	$a_3$
Top tray	0.8490	0.1728	-0.0219
Side draw tray	1.3499	-0.0028	-0.3472
Reboiler	1.2095	0.0411	-0.2506
Prefract. bottom	1.5378	-0.4246	-0.1132
Mid-main bottom	1.4097	-0.2634	-0.1464
Feed heater	1.4992	-0.3488	-0.1505
Reflux heater	1.3939	-0.0164	-0.3775

**Fig. 8. Comparison of measured and calculated temperatures in side draw tray (a) and reboiler (b).**

tion efficiency of the modified distillation system of this study is satisfactory.

For the monitoring and prediction of the temperature variation the autoregressive model was utilized as follows.

$$y(k) + a_1 y(k-1) + a_2 y(k-2) + \dots + a_n y(k-n) = e(k) \quad (1)$$

where  $a_i$ 's are model parameters, and  $e$  is white noise. The  $k$  indicates the step of sampling time. The parameters were computed from the measured temperature using matrix operation. The model parameters for the various temperatures are listed in Table 3. In Fig. 8(a), the comparison of the measured and computed temperatures of side draw tray is shown. The solid line is measured temperature, and the dotted is computed from Eq. (1). The computed temperature variation is very close to the measured one, which indicates the modeling is satisfactory for the monitoring of the column. The same comparison was conducted for the reboiler temperature, and the result is shown in Fig. 8(b). Again, a close prediction of measured temperature was obtained.

The experimental result obtained here shows that the operation of the distillation column in this study was stable and satisfactory. It means that the proposed modification of the FTCDC is appropriate and workable. The vapor flow among the column sections was steady without external compression and liquid flowed by its gravitation. No pump was used here. This experimental study provides the technical verification of the modified FTCDC for the prac-

**Table 4. Comparison of simulation results between the modified FTCDC and conventional distillation system**

Components	FTCDC	Conventional
Tray No.		
1st	10	16
2nd	22	16
Feed [mL/min]	30	30
Pressure [kg/cm <sup>2</sup> ]		
Top	1.0	1.0
Bottom	1.5	1.5
Utilities [kW]		
Condenser	0.83	0.53, 0.42
Reboiler	0.94	0.53, 0.42

tical application of the proposed distillation system. The performance comparison between the modified FTCDC and a conventional distillation system was conducted with simulation for the same feed and total number of trays of the systems. As shown in Table 4, no significant reduction of energy requirement was observed in the modified FTCDC. Because the composition of key component in products is mild, both systems are not energy-intensive processes and no noticeable energy saving is obtained from the modified FTCDC.

## CONCLUSIONS

A modified fully thermally coupled distillation column has been introduced, and its operability was experimentally examined by applying to the separation of methanol, ethanol and *n*-propanol mixture. The modification was aimed to utilize the existing distillation system in the energy-efficient distillation column for the reduction of investment cost. The experiment was conducted in a 4-in sieve tray column. The temperatures of seven different locations in the column were measured to indicate that the column was stably operable. Also, it was proved that neither compressor nor pump is necessary for the vapor and liquid flow between tray sections. The experimental result was compared with that of the HYSYS simulation to demonstrate that a satisfactory separation of the alcohol mixture was experimentally available. The modeling of temperature variation was presented, and it was found that the prediction of the variation was successful.

## ACKNOWLEDGEMENTS

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## NOMENCLATURE

$a$	: model parameters [-]
$B$	: bottom product [kmol/h]
$C1$	: prefractionator [-]
$C2$	: middle main column [-]
$D$	: overhead product [kmol/h]
$E1$	: upper main column [-]

E2 : lower main column [-]  
 F : feed [kmol/h]  
 k : time step [-]  
 L : reflux flow [kmol/h]  
 L<sub>2</sub> : prefractionator liquid flow [kmol/h]  
 L<sub>3</sub> : returning prefractionator liquid flow [kmol/h]  
 S : side product [kmol/h]  
 V : vapor boilup [kmol/h]  
 V<sub>2</sub> : returning prefractionator vapor flow [kmol/h]  
 V<sub>3</sub> : upper main column vapor flow [kmol/h]  
 y : output [-]

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