Dynamic Study of Reactive Dividing Wall Column to Synthesis Dimethyl Ether from Methanol Using Aspen Plus Dynamics

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Abstract. In chemical industry, control system is one of the most important things to ensure product quality and safety. In designing the control system, dynamics models in the form of transfer functions are required. In a complex process the transfer functions are derived from black box models, which the relationships between input and output variables are obtained from the dynamic data. One of the complex processes that can be found in the chemical industry is reactive dividing wall column distillation (RDWC). RDWC can be used to synthesis many chemical compound, one of them is dimethyl ether (DME). DME is chemical compound which has a high economic value for it is widely used as reagent in chemical industries. In distillation column, reboiler duty and reflux rate are usually become manipulated variable (MV) and products purities are usually become controlled variable (CV). So in this study, the MVs of RDWC were reboiler duty and reflux rate, and the CVs were purity of DME at distillate and purity of methanol at side stream outlet. The goals of this study were to find the transfer functions between CVs and MVs, as well as the values of the parameters of the transfer function. To achieve this goals, dynamic simulations of RDWC was carried out by series of step test using Aspen Plus Dynamics. It was found that the response of DME purity to reboiler duty and reflux rate was more sensitive and responsive than the response of methanol to reboiler duty and reflux rate; and the responses of DME and methanol purity to reboiler duty were more sensitive and responsive than their responses to reflux rate. Keywords: reactive dividing wall column, process dynamics, transfer functions, dimethyl ether.

1. Introduction
In a process industry, a control system is important to ensure the quality of product and work safety. To design a control system, process dynamic models are needed in the form of transfer functions. Transfer function is an equation that describes the relationship between the output variable and the input variable. However, the transfer function of complex process is generally difficult to obtain through fundamental models, so in this study, the transfer function between input and output variable was obtained from dynamic data regression.
One of the complex processes that can be found in the chemical industry is reactive dividing wall column (RDWC). RDWC is a process intensification and integration that combines the reactor and dividing wall distillation column in one device. Dividing wall column itself combines two or more columns into one column, so any separation process that need two or more conventional distillation column can be conducted in one dividing wall column. Therefore, RDWC makes multiple separation and reaction process taking place in one column so it is more effective and efficient than the conventional reactor-distillation column set up.

In distillation column, reboiler duty and reflux rate are usually become manipulated variable (MV) or input and products purities are usually become controlled variable (CV) or output [1]. So in this study, the MVs of RDWC were reboiler duty and reflux rate, and the CVs were purity of DME at distillate and purity of methanol at side stream outlet.

The goals of this study were to find the transfer functions between CVs and MVs, and the value of the transfer functions parameters. To achieve this goal, the RDWC was made with Aspen Plus and validated with existing literature data. After validation, a dynamic simulation was done by series of step test using Aspen Plus Dynamics, so dynamic data was obtained. The data was then regressed using a variety of transfer function models.

2. Method

2.1. Model validation

Model validation was the first step in this study. This validation was done using Aspen Plus V8.8 with UNIQUAC-RK as property methods. Validation was done by comparing literature data (Table 1 and Table 2) with the created model. Literature data was derived from [6]. The model of RDWC is shown in Figure 1.

![Figure 1. RDWC Model on Aspen Plus](image)

| Parameters                  | Value |
|-----------------------------|-------|
| Number of stages            | 35    |
| Reactive stages              | 8-31  |
| Feed stage                  | 8     |
| Wall position                | 8-31  |
| Distilat to Feed ratio      | 0,25  |
| Reflux ratio (kmol/kmol)     | 2,45  |
| Condenser Pressure (bar)     | 10    |

Table 1. Column specification [6]
Table 2. Feed specification [6]

| Parameters                          | Value |
|------------------------------------|-------|
| Feed flow rate (kmol/h)            | 9     |
| Feed temperature (°C)              | 25    |
| Feed pressure (bar)                | 10    |

Methanol in feed stream becomes DME through dehydration reaction with γ-alumina catalyst. The reaction kinetic is [6]:

$$2 \text{CH}_3\text{OH} \leftrightarrow \text{CH}_3\text{OCH}_3 + \text{H}_2\text{O} \quad (1)$$

$$r = kW_{cat}[\text{MeOH}]^m[\text{H}_2\text{O}]^n \quad (2)$$

$$k = A \exp\left(-\frac{E_a}{RT}\right) \quad (3)$$

where:
- $A$: Arrhenius factor (5.19 x 10⁹ m³.kg-cat⁻¹.s⁻¹)
- $E_a$: Activation energy (133.8 kJ.mol⁻¹)
- $m = 1.51$
- $n = -0.51$

2.2. Dynamic simulation

Dynamic simulation is done by changing the MVs value within the range of ± 5-10%. The changes in purity of dimethyl ether and methanol (CVs) due to the changes MVs were then observed. The MVs-CVs data was then regressed to obtain appropriate transfer functions.

3. Results and discussion

3.1. Model validation

Table 3 shows that the purity of the DME at distillate is 99.99%, so it was the same as in the literature. The value of reboiler duty to achieve this DME purity was almost the same as in the literature. The difference may be because of the difference in vapor-liquid split ratio. In this study, vapor-liquid split ratio was 0.31, while there was no information in literature data. The water purity at bottom was also almost the same as in the literature. Therefore, this model validation can be said to have succeeded.

Table 3. Validation Results

| Parameters              | Literature [6] | Model   |
|-------------------------|----------------|---------|
| DME purity (%)          | 99.99          | 99.99   |
| Water purity (%)        | 99.99          | 99.92   |
| Reboiler duty (kW)      | 58.7           | 60.83   |

3.2. Dynamic simulation

In dynamic simulation, the changes in DME purity at distillate and methanol purity at side stream due to the series of step test in reboiler duty (Qreb) and reflux rate (Rf) were observed. The initial value of reboiler duty was 60.83 kWh. From this initial value, series of step test were conducted within the range of ± 5-10%. The dynamic data is shown in Figure 2. It was found that when reboiler duty increases, the purity of dimethyl ether in distillate was decreased. It is because when reboiler duty increases, there were much more evaporation of the other components (water and methanol). These components went to the distillate output stream and were mixed with DME so the purity of DME was decreased.
The data was then regressed with MATLAB software so it was found that the transfer function between reboiler duty and DME purity was:

$$G_{p1} = \frac{-6.973 \times 10^4 s - 5.612 \times 10^4}{s^2 + 4.485 \times 10^4 s + 9141}$$  \hspace{1cm} (4)$$

The form of $G_{p1}$ was the best transfer function to describe the input-output data profile. This argument is supported by The Time Response Comparison of $G_{p1}$ with the data (Figure 3) and the $R^2$ value of 0.9659. From this transfer function, the gain value was calculated by dividing numerator constant ($-5.612 \times 10^4$) with denominator constant (9141) resulting in the value of -6.14. The negative sign indicates that the output (DME purity) was inversely proportional to the input (reboiler duty). The high order time constant was calculated as square root of 9141, resulting in the value of 0.01 h.

![Figure 2. Input-output dynamics: DME purity vs Reboiler Duty (Qreb)](image)

With the same method, the transfer functions for the other input-output were derived. The results are shown in equation (5) to (7), while the gains and the high order time constants are shown in Table 4.

$$G_{p2} = \frac{-1.326 \times 10^5 s^3 - 1.261 \times 10^6 s^2 - 0.684 \times 10^6 s - 5.151 \times 10^6}{s^5 + 429.7 s^4 + 1.066 \times 10^5 s^3 + 8.511 \times 10^5 s^2 + 4.559 \times 10^6 s + 1.369 \times 10^6}$$  \hspace{1cm} (5)$$

$$G_{p3} = \frac{0.9296 s^3 + 9.12 s^2 + 19.19 s + 8.906}{s^4 + 2111.1 s^3 + 1.873 \times 10^5 s^2 + 2.710 \times 10^5 s + 6764}$$  \hspace{1cm} (6)$$

$$G_{p4} = \frac{0.003479 s^3 + 0.768 s^2 + 0.2576 s + 0.03399}{s^4 + 2116.9 s^3 + 9115.2 s^2 + 4372 s + 1600}$$  \hspace{1cm} (7)$$

$G_{p2}$ is transfer function between methanol purity and reboiler duty. $G_{p3}$ is transfer function between DME purity and reflux rate, while $G_{p4}$ is transfer function between methanol purity and reflux rate.
Figure 3. Time response comparison of \( G_{p1} \) with the changes of DME purity due to series of step test on reboiler duty

The gain for \( Q_{reb} - \) Methanol purity was negative. It means reboiler duty was inversely proportional to methanol purity at side stream outlet. There are two reasons that may be causing this result. The first reason is the increasing reboiler duty will move the reaction equilibrium to the products since it is endothermic reaction. The second reason is the increasing reboiler duty will also increase the evaporation of bottom products. These will reduce the purity of methanol at side stream.

Table 4. Gain and high order time constant result

| Input-output       | Gain (K)   | High order time constant (\( \tau \)) |
|--------------------|------------|--------------------------------------|
| \( Q_{reb} - \) DME purity | -6.14      | 0.01                                 |
| \( Q_{reb} - \) Methanol purity | -3.76      | 0.06                                 |
| \( R_f - \) DME purity     | \( 1.32 \times 10^{-3} \) | 0.11                                 |
| \( R_f - \) Methanol purity | \( 2.12 \times 10^{-5} \) | 0.16                                 |

The gain of \( R_f - \) DME purity and \( R_f - \) Methanol purity were positive. It means reflux rate is proportional to DME and methanol purity. This is because the greater the reflux rate, the more contact between liquid and vapor in the column, so DME and Methanol purity are increase.

The greater the gain, regardless of its sign (negative/ positive), the output is more sensitive to input change. So it can be seen in Table 4 that a change in reboiler duty or reflux rate will make greater change in DME purity compare to methanol purity. It is because reboiler and reflux stream were attached in the main column that discharges DME as distillate, while methanol was discharged at side column (right side after the wall). This makes the DME purity was affected directly to the change in reboiler duty and reflux rate.

This result was also synchronous with the calculated high order time constant (\( \tau \)). The greater the \( \tau \), the output is less responsive to input change. From Table 4, it is seen that if there was a change in reboiler duty or reflux rate, the DME purity responded faster than methanol purity. This was also because the DME purity was affected directly to the change in reboiler duty and reflux rate.

The DME and methanol purity are both more sensitive and more responsive to a change of reboiler duty than to a change of reflux rate.
This is because of the change in reboiler duty affects temperature profile in the column more significant than the change in reflux rate, while the change in reflux rate affects concentrations profile in the column more significant than the change in reboiler duty. According to equation (2) and (3), it can be derived that the change in temperature will affect reaction rate much more significant than the change in concentrations. The reaction rate surely affects DME and methanol purity. Therefore, the responses of DME and methanol purity to reboiler duty were more sensitive and responsive than their responses to reflux rate.

4. Conclusions
The following conclusions may be derived from the study:
- Reboiler duty was inversely proportional to DME and methanol purity.
- Reflux rate was proportional to DME and methanol purity.
- The response of DME purity to reboiler duty and reflux rate was more sensitive and responsive than the response of methanol to reboiler duty and reflux rate.
- The responses of DME and methanol purity to reboiler duty were more sensitive and responsive than their responses to reflux rate.

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