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Identification of a Binary Distillation Column Using Pulse Testing

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Abstract

The binary system of methanol-tert-buthanol was studied at atmospheric pressure in a 53-tray, 35 mm inside diameter sieve tray distillation column. The column had a vertical thermosiphon reboiler and an overhead condenser. During the continuous distillation, the effect of the reflux flow rate and vapour flow rate to the column as rectangular pulse input variables were investigated in the frequency domain. The experimental rectangular pulse input x(t) and output y(t) of the column were entered into a Pulse Test Program. The real and imaginary roots, log modulus phase angles and steady state gains (K) were calculated by the program. Then, Bode plots were drawn. The time constants and time delays were calculated from the Bode plots. The open-loop transfer functions of the top and feed plate were derived. The transfer functions and initial parameters of the column were entered into a commercial simulation program. The experimental and simulation results were compared.

Key Words: Distillation column, frequency domain analysis, dynamic behaviour

Darbe Testi Kullanılarak İkili bir Destilasyon Kolonunun Dinamik Davranışının Tanımlanması

Özet

Metanol -ter -butanol ikili karışımının sürekli destilasyonu 53 platolu, 5 mm çapındaki delikli platolu bir destilasyon kolonunda atmosferik basınçta yapılmıştır. Destilasyon kolonu, bir adet termosifon tipi kazan ve bir adet tepe soğutucusundan ibarettir. Kararlı halde (steady-state) çalışan kolonun reflüks ve buhar akışlarına dikdörtgen puls (rectangular pulse) tarzında istenmeyen girdi değişimi uygulanmış ve kolonun bu girdilere verdiği cevap incelenmişitr. Kolona uygulanan girdi değişimleri x(t) ve kolonun verdiği cevap değerleri y(t), Puls Test Programına girilmiştir. Puls Test Programı çıktılarından gerçek ve sanal kökler, log modulu, faz açısı ve kararlı hal proses kazancı (K) değerleri hesaplanmıştır. Daha sonra Bode çizimleri yapılmş ve bu çizimlerden zaman sabit (τ) ve ölü zaman (θ) parametreleri hesaplanarak kolonun açık devre (open-loop) transfer fonksiyonları türetilmiştir. Transfer fonksiyonları ve kolonun başlangıç parametreleri ticari bir simülasyon programına girilmiştir. Simülasyon sonuçları ile deneysel değerler karşılaştırılmıştır.

Anahtar Sözcükler: Destilasyon kolonu, frekans cevap analizi, dinamik davranış

1. Introduction

Distillation is the most commonly used separation process in the chemical and petroleum industries (Kapoor et al., 1986 Skogestad et al., 1990). In 1992, Darton presented his estimates of the world-wide throughput of distullation columns.
Oil refining

3.7 billion tonnes per year

Chemicals and petrochemicals

130 million tonnes per year

Natural gas processing

1.4 billion tonnes per year

Taking the current price of crude oil at $18 per barrel, this means that in financial terms the throughput of distillation columns is worth at least $524 billion/annum (Porter, 1995). An important requirement in the design of automatic control systems for distillation columns is a knowledge of the dynamic behaviour of the system (Wahl et al., 1979; Semino et al. 1997). In a previous study (Kırbaşlar et al., 194), the effect of feed concentration and reflux flow rate as step variables were investigated in the time domain. The parameters were calculated from the process reaction curve by the methods described by Harriot and Smith. This research extends the treatment to temperature responses of the vapour flow rate and reflux flow rate with pulse changes in the frequency domain.

The response of a distillation column to disturbances in any of its stream variables (flow rate, composition or energy), can in principle, be established by recording the mass and energy balances of every plate, condensers and reboiler and then solving the resultant set of simultaneous differential equations (Distefano et al., 1967; Edwards et al., 1977; Jacobsen, 1997).

In practice, the experimental approach is sometimes used when the process is thought to be too complex to model and the values of some parameters can be calculated from steady state plant data, but some parameters must be found with dynamic tests (Luyben, 1973; Seborg, 1989).

The purpose of this research was to calculate the time constant, time delay and pocess gain of the distillation column in the frequency domain and to derive open-loop transfer functions from the column parameters.

2. Theoretical Framework

In recent years a number of parameter estimation techniques have been reported (Chen et al., 1979; Harrison et al., 1974; Johnson et al., 1971; Sundaresan et al., 1978; Zhou et al., 1995; Weigand et al. 1972; Andersen et al. 1989; Huang and Huang., 1993). Four methods have been proposed for estimating the dominant time constant and time delay of a given process from knowledge of its moment, s-plane, frequency or transient response data.
These methods can be classified as follows:

1) time domain fitting

2) frequency domain fitting

3) s-domain fitting of the form G(s)

4) moment analysis

Comparisons between these techniques and their applications have also been discussed with the aid of data largely based on experimental or simulated dispersion models (Sundaresan et al., 1978; Li and Lee, 1996).

3. Frequency Domain Method

The advantages of frequency response methods for the dynamic analysis of physical systems are well known. The classical method of obtaining a frequency response experimentally is based on its definition. While the system input x(t), is varied sinusoidally at frequency ω; the system ouput y(t), is measured simultaneously.

4. The Use of a Pulse Test To Obtain Frequency Response Data

The pulse testing method has proved satisfactory in many plant applications (Seborg et. al. 1989; Li and Lee, 1996) because it is relatively easy to implement; it avoids the necessity of performing many different tests to obtain process input-output data at different frequencies. In conducting a pulse test, the input variable is changed from its steady-state value in a pulselike manner (Figure 1). The shape

of this pulse does not necessarily match any prescribed function. Generally, the process output will respond in a similar, pulselike fashion, although over a longer duration. Since an input pulse contains a range of frequencies, in effect the process is forced simultaneously by each of these frequency components. Hence, although additional computations are required to generate the frequency response from input, output data, theoretically a single experiment can yield the entire frequency representation of the process. In practice, several pulse tests might have to be performed to obtain a more complete data base.

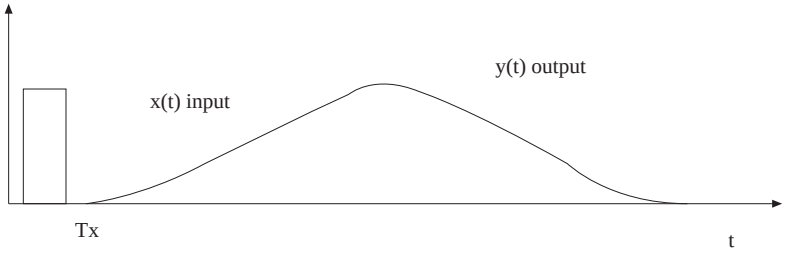

Figure 1. Rectangular pulse input and output curve

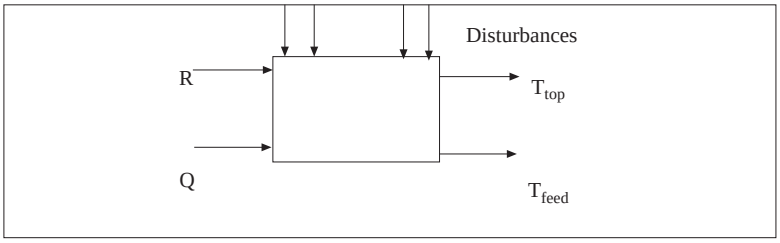

Figure 2. Block diagram of a distillation column

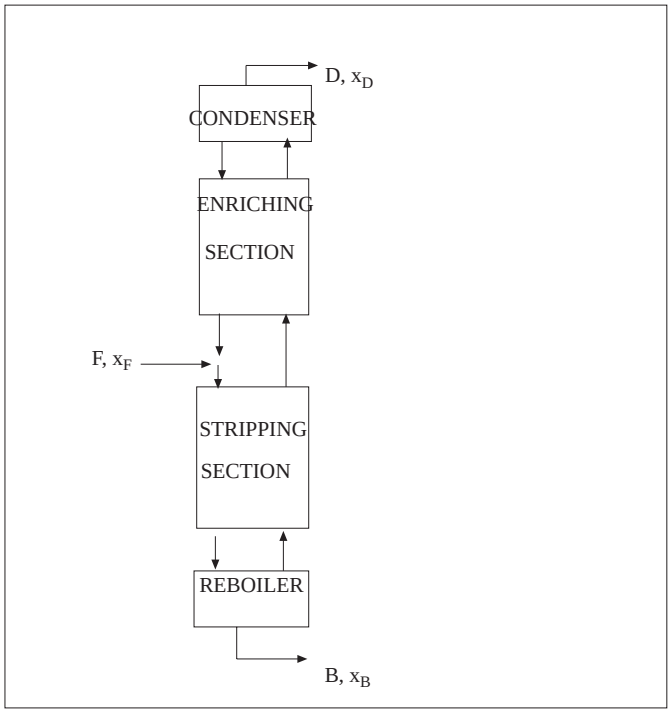

Figure 3. Schematic diagram of the distillation column

A pulse test is performed first by permitting the process to come to a steady state. A closed pulse, that begins and ends at the steady-state operating value, is then introduced as an input variable in the process. The time at which the input variable first deviates from its steady-state values is specified as zero time. Normally the final time is chosen to be when both the input and output pulses have returned to and remain at their previous steady state values.

Pulse testing has been used in a number of studies in the literature (Hougen, 1979; Clement and Schnelle, 1963; Hwang et al. 1994). Pulse testing retains many of the advances of direct sine-wave testing. The pulse testing metod involves conversion of pulse data to frequency from throughout a numerical Fourier transformation. The efficiency of the Fourier analysis and its usefulness in estimating the best model parameters have been discussed by a number of authors (Johnson et al. 1971) Luyben, 1987; Sundaresan et al. 1978). The estimation procedure involves maching experimental data to the assumed model in the frequency domain.

Assume that an optional pulse-type signal x(t) is the input of a process and y(t) is the corresponding output (Figure 1). The transfer function of the process is defined as follows:

$$G(s) = \frac{Y(S)}{X(S)} \tag{1}$$

Using the definition of the Laplace transformation,

$$G(s) = \frac{\int_0^\infty y(t)e^{-st}dt}{\int_0^\infty x(t)e^{-st}dt} \tag{2}$$

Because y(t) and x(t) are deviation variables, the numerator and denominator integrals of equation 2 need to be evaluated over the output pulse duration T_y and the input pulse duration T_x since y and x are equal to zero everywhere else. Then, substituting iω for s in equation 2 gives:

$$G(iw) = \frac{\int_0^{T_y} y(t)e^{-i\omega t}dt}{\int_0^{T_x} x(t)e^{-i\omega t}dt} \tag{3}$$

The numerator is the Fourier trasformation of the time function y(t). The denominator is the Fourier transformation of the times function x(t). Applying the identity $e^{-i\omega t} = \cos\omega t - i\sin\omega t$ to both integrals in equation 3 gives equation 4. When equation 4 is subject to algebraic reduction, it gives the following basic equations. Then, these equations are used for obtaining frequency response data from pulse tests.

$$G(i\omega)=\frac{\int_0^{T_y} y(t)\cos(\omega t)dt - i\int_0^{T_y} y(t)\sin(\omega t)dt}{\int_0^{T_x} x(t)\cos(\omega t)dt - i\int_0^{T_x} x(t)\sin(\omega t)dt} \tag{4}$$

$$G(i\omega)=\frac{A-iB}{C-iD}=\frac{(AC+BD)+i(AD-BC)}{C^2+D^2} \tag{5}$$

$$G(i\omega) = ReG(i\omega) + iImG(i\omega) \tag{6}$$

where

$$A = \int_0^{T_y} y(t)\cos(\omega t)dt \tag{7a}$$

$$B = \int_0^{T_y} y(t)\sin(\omega t)dt \tag{7b}$$

$$C = \int_0^{T_x} x(t)\cos(\omega t)dt \tag{7c}$$

$$D = \int_0^{T_x} x(t)\sin(\omega t)dt \tag{7d}$$

The problem is reduced to being able to evaluate the integrals A, B, C and D given in equations (7a) through (7d) for known functions y(t) and x(t). The integrations are with respect to time between the definite limits of zero and the times that the experimental time functions go to zero, T_y for y(t) and T_x for x(t).

A numerical values of frequency ω is selected. The integrations are performed on a computer, giving one point on the frequency response curves (Luyben, 1973). Then, frequency is changed and the integrations repeated, using the same experimental time functions y(t) and x(t) but a new value of frequency ω. Repeating for frequencies over the range of interest gives the complete G(ω). The y(t) and x(t) data are used over and over again.

5. Transfer Funciton Model and Block Diagram Analysis of the Column

A mathematical model is usually required for the desing of a control system. Since most chemical processes are self-regulated, do not oscillate, and contain some amount of time delay, the second-order-plus-dead-time model can be presented in terms of a single time constant and a damping ratio as

$$G(s) = \frac{Y(s)}{X(s)} = \frac{Ke^{-\theta s}}{\tau^2 s^2 + 2\tau\zeta s + 1} \tag{8}$$

or in terms of two time constants as

$$G(s) = \frac{Y(s)}{X(s)} = \frac{Ke^{-\theta s}}{(\tau_1 s + 1)(\tau_2 s + 1)} \tag{9}$$

Mathematically, these two forms are equivalent but the equation (8) form is preferable for underdamped cases and can be used for all τ and ζ. The equation (9) form can be used only for the overdamped case (Huang, 1982).

Considering the distillation column control problem shown in Figure 2, since there are two controlled variables and two manipulated variables, four process transfer functions are necessary to completely characterise the process dynamics:
Multiple-input, multiple-output process (2×2)

$$G_{p11}(s) = \frac{T_{Top}(S)}{R(s)} G_{p12}(s) = \frac{T_{Top}(S)}{Q(s)}$$
$$G_{p21}(s) = \frac{T_{Feed}(S)}{R(s)} G_{p22}(s) = \frac{T_{Feed}(S)}{Q(s)} \tag{10}$$

The transfer functions in equation 10 can be used to determine the effect of a change in either R or Q. From the principle of superposition (Seborg, 1989; Skogestad, et al. 1990; Schei, 1992), it follows that simultaneous changes in R and Q have an additive effect on each controlled variable:

$$T_{Top}(S) = G_{p11}(s)R(s) + G_{p12}(s)Q(s) \tag{11}$$

$$T_{Feed}(S) = G_{p21}(s)R(s) + G_{p22}(s)Q(s) \tag{12}$$

These input-output relations can also be expressed in vector-matrix notation as

$$T(s) = G_p(s)M(s) \tag{13}$$

Where T(s), (output) and M(s), (input) are vectors with two elements,

$$T(s) = \begin{bmatrix} T_{Top}(S) \\ T_{Feed}(S) \end{bmatrix} M(s) = \begin{bmatrix} R(s) \\ Q(s) \end{bmatrix} \tag{14}$$

and G_p(s) is the process transfer function matrix,

$$G_p(s) = \begin{bmatrix} G_{p11}(s) & G_{p12}(s) \\ G_{p21}(s) & G_{p22}(s) \end{bmatrix} \tag{15}$$

The transfer functions characterising the column dynamics were established by pulse testing. The parametrs of the assumed scond-order-plus-time-delay transfer function were determined from the transient data (Andersen and White, 1970; Seborg, 1989).

6. Experimental

All tests were made with the methanol-tert-buthanol system at a total pressure of 760 mmHg. The methanol and tert-buthanol (Merck Co.) used were of reagent grade. The transient response of the system was examined when it was subjected to two different types of disturbance change, reflux flow rate to the top plate and reboiler heat duty. Rectangular pulse changes of the input perturbations were applied and the magnitude and direction of these changes were varied.

The experimental distillation system consisted of a 35 mm diameter column made from two glass segments. There were 53 sieve trays. The tray spacing was 31 mm. The column with reboiler and condenser was 3,000 mm high (Figure 3). The feed flow was fed into the 28th plate, above the reboiler. One resistance thermometer, (Pt-100) was installed in the feed plate to measure its temperature. The saturated vapours, coming from the column, were partially condensed by the condenser installed at the top of the column. The condensed liquid was separated into two parts in chosen ratios by a special solenoid valve. The first part left the column and then passing through the second condenser; was cooled to room temperature and fed into the accumulation tank as distillate. The second part of this liquid was fed back into the column as reflux. The reflux flow rate was controlled manually by a reflux timer on the control unit. In addition, one resistance thermometer, was installed in the top of the column to measure its temperature. The thermosiphon reboiler with a capacity of 1500 mL was heated by a 1 kW special quartz heater. It was possible to regulate the energy supplied to the heater with a special variac and in this way the desired vapour velocity could be obtained. Similarly, one resistance thermometer was installed in the reboiler of the column to measure its temperature. The feed mixture was fed in and the reboiler product was withdrawn with special dosage pumps (Prominent).

7. Operating Procedure

The column was operated initially under steady-state conditions until all temperatures, pressures, flow rates and compositions were constant. To eliminate uncertainties in evaluating steady-state parameters, a number of modified pulse input runs were carried out. Temperatures were measured with

resistance thermometers. The compositions were analysed by gas chromatography (Hewlett-Packard Model 6890) equipped with a capillary column. The chromatograph was operated with the following parameters:
Detector : FID, operating at 553 K
Column : Internal diameter 0.25 mm; 30,000 mm long with an HP-INNOWax Poyethylene Glycol Capillary column.
Injection block: 473 K
Gas flow rates:
Carrier gas(N_2):45.9 mL/min

Hydrogen : 33 mL/min

Air : 400 mL/min
Oven
Initial : 323 K

Heating ramp : 5 K/min

Post run : 373 K

Expect for the specific disturbance (heat supply to the reboiler or reflux flow rate to the top plate) introduced, the initial steady-state conditions were maintained during the transient period. The variables held constant were feed rate, feed composition, feed temperature, column pressure, top product rate and bottom product rate. Average values for the steady-state variables employed in all of the runs are given in Table 1. The vapour rate was changed as a rectangular pulse by changing the heat supply to the reboiler. The reflux flow rate was changed as a rectangular pulse by the reflux switch on the control panel.

The top plate, feed plate and reboiler temperatures were measured every 30 seconds for first 10 minute of the transient period; then every 1 minute for the next 10 minutes and finally every two minutes. The experimental responses of the first 4 runs against reboiler duty (vapour flow rate) rectangular pulse changes are shown Figs. 4-7. The experimental response of the last 4 runs against reflux flow rate rectangular pulse changes are shown in Figs. 8-11.

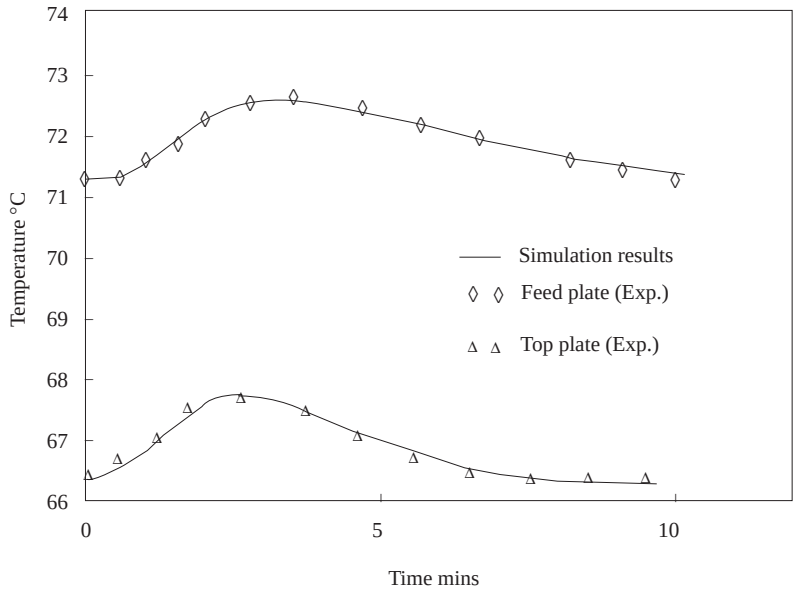

Figure 4. Experimental and simulation temperature responses of top and feed plate. The heat supply to the reboiler was increased from 321 kJ/h to 1473 kJ/h for 1.5 min. (Run 1).

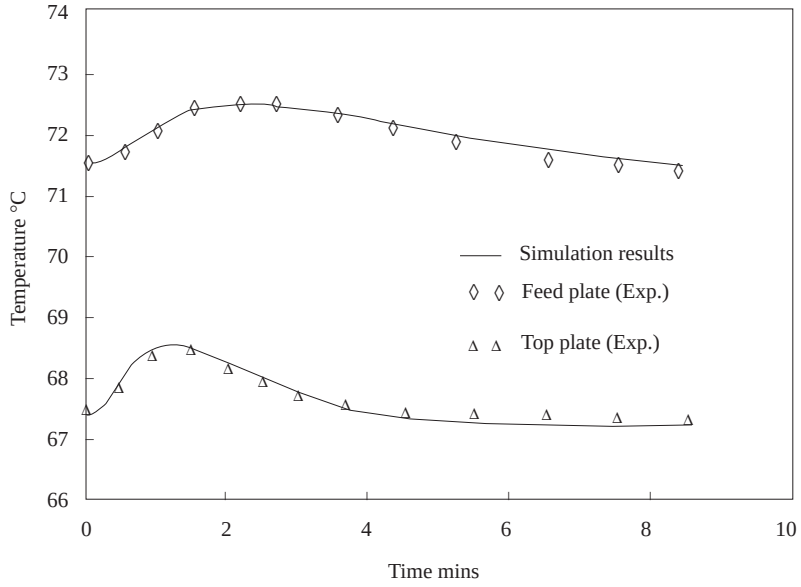

Figure 5. Experimental and simulation temperature responses of top and feed plate. The heat supply to the reboiler was increased from 455 kJ/h to 2121 kJ/h for 0.5 min. (Run 2).

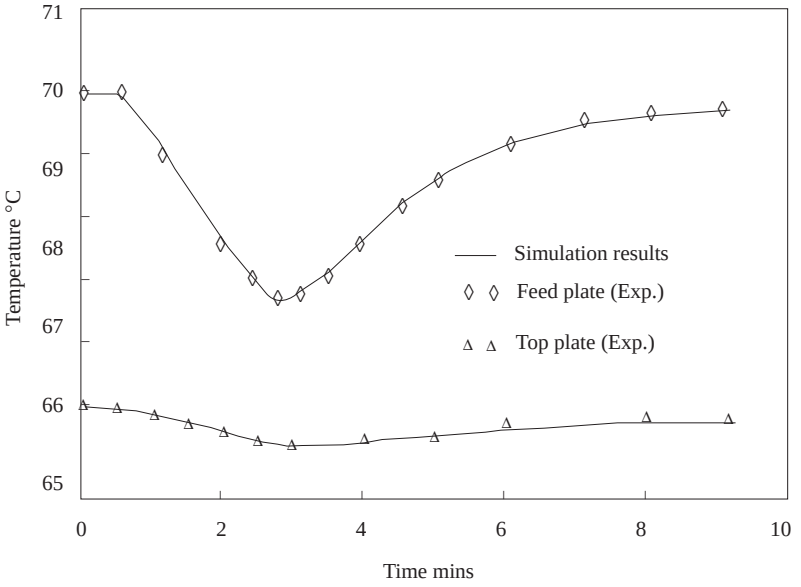

Figure 6. Experimental and simulation temperature responses of top and feed plate. The heat supply to the reboiler was increased from 530 kJ/h to 105 kJ/h for 2.0 min. (Run 3).

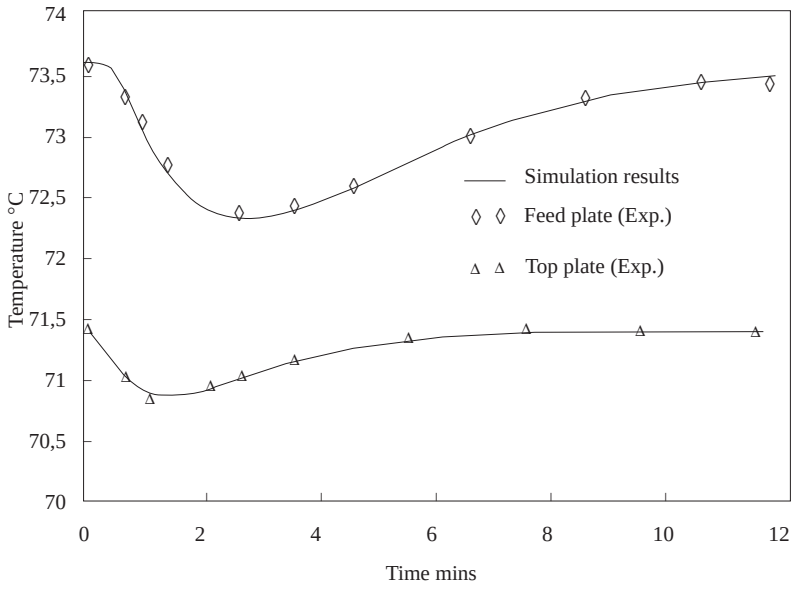

Figure 7. Experimental and simulation temperature responses of top and feed plate. The heat supply to the reboiler was increased from 530 kJ/h to 105 kJ/h for 1.5 min. (Run 4).

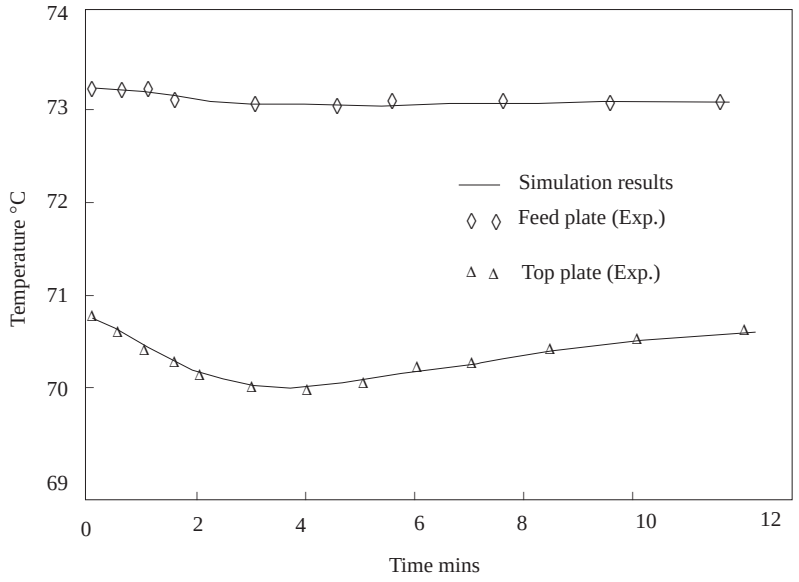

Figure 8. Experimental and simulation temperature responses of top and feed plate. The reflux flow to the top plate was increased from 6.88 mole/h to 17.21 mole/h for 1.25 min. (Run 5).

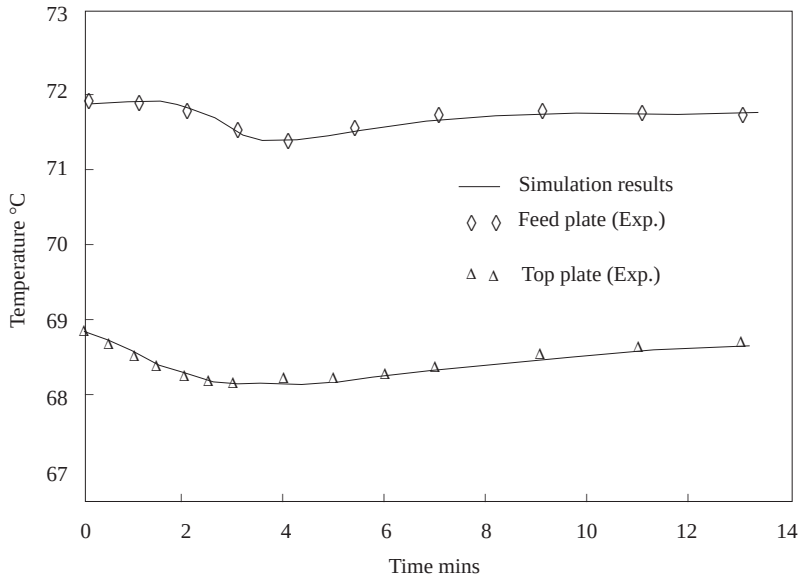

Figure 9. Experimental and simulation temperature responses of top and feed plate. The reflux flow to the top plate was increased from 7.85 mole/h to 19.64 mole/h for 1.5 min. (Run 6).

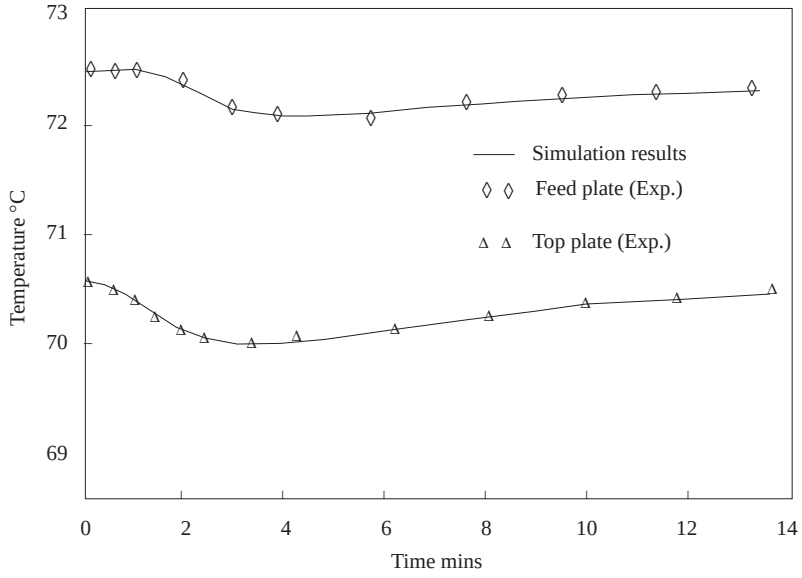

Figure 10. Experimental and simulation temperature responses of top and feed plate. The reflux flow to the top plate was increased from 5.08 mole/h to 15.23 mole/h for 1.5 min. (Run 7).

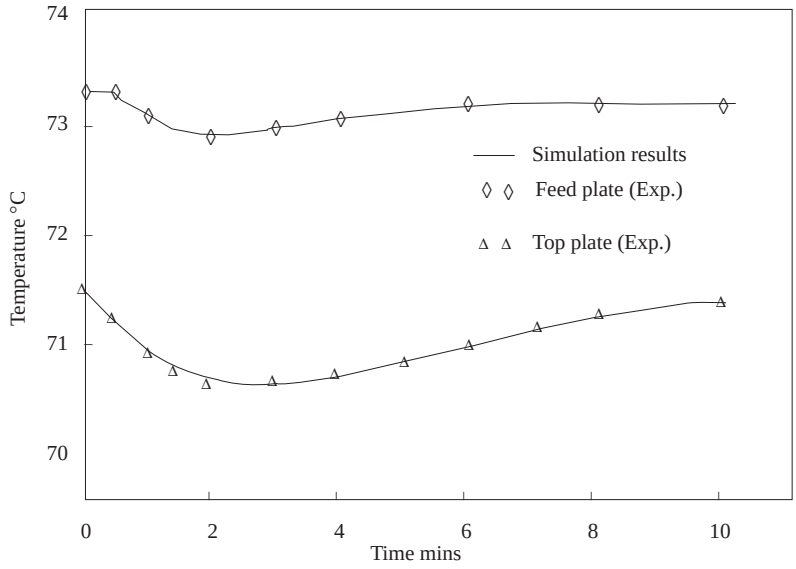

Figure 11. Experimental and simulation temperature responses of top and feed plate. The reflux flow to the top plate was increased from 6.78 mole/h to 20.35 mole/h for 1.0 min. (Run 8).

8. Results and Discussion

The open loop dynamics were investigated by applying rectangular pulse changes in reflux flow rate and reboiler heat duty. In order to estimate the process time constants and to show the dynamic behavior of the column, a number of rectangular pulse tests were performed. The application time of the rectangular pulse was changed from 0.5 minute to 2 minutes. In the first run, heat supply to the reboiler was increased from about 321 kJ/h to 1473 kJ/h for 1.5 minutes and then returned to its initial value. As a result, the vapour flow rate changed from the bottom to the top of the column. In the last run, the reflux flow rate was increased from 5.702 mole/h to 17.06 mole/h for 1.0 minute, then returned to its initial value. Consequently, the reflux flow rate changed from the top plate to the reboiler.

A reasonable number of points were selected from experimental rectangular pulse input, x(t) and output data, y(t) then entered into the Pulse Test Program. The real and imaginary roots, log modulus and phase angles were calculated by the program. Steady state gains (K) were calculated by the Pulse Test Program and are given in Table 2. The flow chart of the Pulse Test Program is shown in figure 12. Bode plots were drawn from the output of the program. Then, the time constant (τ) and time delay (θ) were calculated from Bode plots (Seborg et al. 1989). The time delays are given in Table 2. The time constants (τ) of the top plate and feed plate were normalised. Then the open loop empirical model of the column was developed as shown in equation 16.

$$\begin{bmatrix} T_{Top}(S) \\ T_{Feed}(S) \end{bmatrix} \begin{bmatrix} \frac{-1.3e^{-0.2s}}{(3.45s+1)(2.05s+1)} \\ \frac{-1.0e^{-0.75s}}{(3.23s+1)(2.00s+1)} \end{bmatrix} \begin{bmatrix} \frac{-0.08e^{-0.35s}}{(1.43s+1)(0.92s+1)} \\ \frac{-0.2e^{-0.6s}}{(3.57s+1)(1.02s+1)} \end{bmatrix} \begin{bmatrix} R(s) \\ Q(s) \end{bmatrix} \quad (16)$$

Experimental and model Bode plots for the top plate of the Run 2 are shown in Figure 13. It is shown that the Bode plots obtained from experimental data and model transfer were in good agreement (Figure 13). The top tray and feed tray of the column behaved as a second-order-time-delay process. The general form of the transfer function is;

Table 1. Operating Data of Dynamic Experiments on the Distillation Column.

Run number	1	2	3	4	5	6	7	8
Pulse change								
$1^{st}(Q_{1,kJ/h})$;(R_1 mole/h)	321	455	530	530	6.88	7.85	5.08	6.78
$1^{nd}(Q_{2,kJ/h})$;(R_2 mole/h)	1473	2121	105	105	17.21	19.64	15.23	20.35
Pulse application time (min.)	1.5	0.5	2.0	1.5	.1.25	1.5	1.5	1.0
Transient response time (min.)	10	10	8	6	13	11	15	10
Feed								
Temp.°C	64.5	64.5	64.5	64.5	64.5	64.5	64.5	64.5
Flow rate	7.978	7.978	7.978	7.978	7.978	7.978	7.978	7.978
(mole/h)	0.500	0.500	0.500	0.500	0.500	0.500	0.500	0.500
Comp. (mole frac. of methanol) x_F								
Reflux ratio (R)	2	2	2	2	2	2	2	2
Vapour rate (mole/h)	5.92	13.58	9.61	5.59	11.64	12.70	6.99	12.32
Overhead prod.								
Flow rate	1.97	4.53	3.21	2.86	3.88	4.23	2.33	4.11
(mole/h)								
	0.999	0.979	0.999	0.959	0.950	0.975	0.770	0.946
Comp. (mole frac. of methanol)X_D								
Bottom prod.	5.91	3.35	4.67	5.02	4.00	3.65	5.55	3.77
Flow rate								
(mole/h)								
Comp. (mole frac. of methanol) x_F	0.595	0.333	0.648	0.403	0.259	0.323	0.129	0.194

$$G(s) = \frac{Ke^{-\theta s}}{(\tau_1 s + 1)(\tau_2 s + 1)} \tag{17}$$

Since the reboiler volume was greater than any tray volume, the change in reboiler temperature could not be measured. Consequently, the reboiler parameters could not be calculated.

The transfer function and initial steady state parameters of the top and feed plates were entered into a commercial simulation program. The same rectangular pulse changes in reflux flow rate and in heat duty of the reboiler were implemented both on the column and simulation. The experimental and simulation results were compared for each run and shown on the same figures (Figure 4-11). The general block diagram of the simulation program is shown in Figure 14. The top and feed plate temperatures are well modelled by the second-order-plus-time-delay model.

Further work will be focused on the experiment and simulation of the PID controlled closed loop behaviour of the column.

Table 2. Steady state gains and time delays of the top and feed trays

Run	1	2	3	4	5	6	7	8
Gain (top tray)	0.008	0.010	-0.003	-0.250	-1.800	-1.500	-1.300	-1.000
Gain (feed tray)	0.015	0.028	-0.012	-0.080	-0.543	-0.543	-1.000	-0.250
Time delay (top tray).	0.42	0.35	0.54	0.41	0.28	0.25	0.18	0.21
Time delay (feed tray)	0.65	0.54	0.61	0.54	1.64	1.60	1.41	0.81

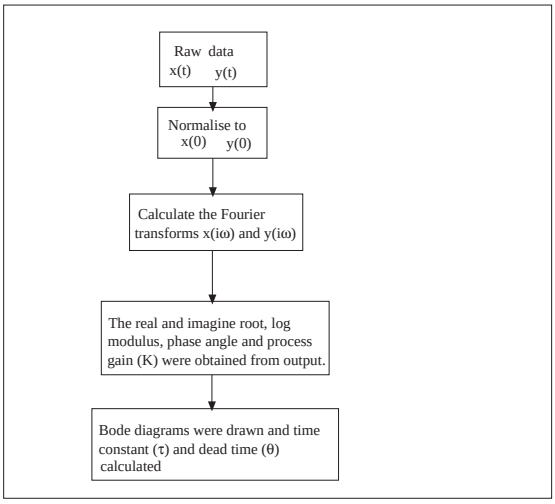

Figure 12. Flow chart of data processing involved in frequency method of response data analysis

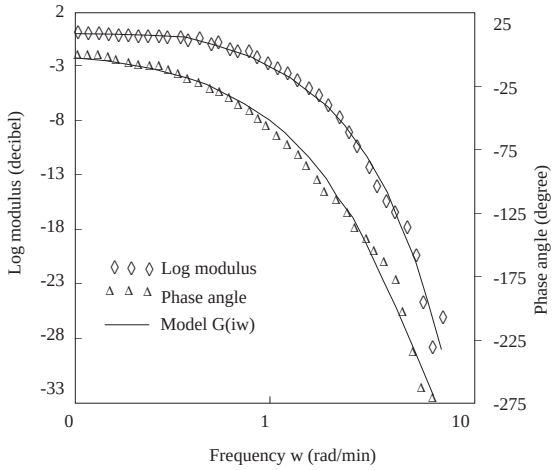

Figure 13. Experimental and model Bode plots of the top plate for Run 2.

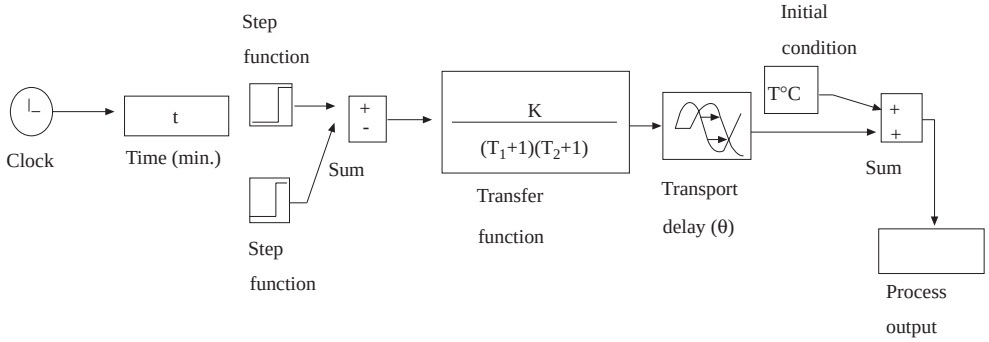

Figure 14. Simulation block diagram of the distillation column.

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Notations

B	bottom product flow rate,mole/h
D	top product flow rate, mole/h
F	feed flow rate, mole/h
G(iω)	experimental frequency, domain transfer function
i=	$\sqrt{-1}$; complex number
Im	imaginary part of a complex number
K	process gain for heat input °C/kJ/h, and for reflux input °C/mole/h
Q	heat duty to the reboiler, kJ/h
R	reflux flow rate, mole/h
Re	real part of a complex number
s	Laplace transform variable
w	frequency, radians/minute
x(t)	experimenatl process input
x_B	bottom product mole fraction
x_F	feed mole fraction
x_D	top product mole fraction
y(t)	experimental process output
θ	time delay, min.
τ	time constant

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