Commissioning and Empirical Modeling of the Dynamic Behaviour of a Closed-Cycle Distillation System

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ABSTRACT

Undergraduate chemical engineering students often lack practical experience with real-world processes, equipment, and control strategies encountered in the industry. This research aims to bridge this educational gap by commissioning and modeling a closed-cycle distillation system at the Process Modeling and Control (PMC) laboratory, so that it can be used by the Department as a hands-on learning platform. The project involved completing the physical setup, calibrating instrumentation, and configuring a National Instruments Data Acquisition (DAQ) system. Experiments were designed and conducted to obtain data for developing First-Order-Plus-Dead-Time (FOPDT) models, which approximate the dynamic behavior of the system during startup. Analysis of these models provided valuable insights into the system's response to operational changes. Although some operational parameters, such as the minimum reboiler level for safe operation, were identified, the focus of the research was in modeling and understanding the system dynamics during startup. The study encountered challenges with controlling both reflux and feed, as the original configuration did not allow flow rates to be set low enough, and the small heat buffer of the trays led to rapid cooling when they were introduced. This limitation was addressed by improving the control mechanisms, enabling both flows to be managed at much lower rates, thereby expanding their operational window at the lower bound. Additionally, the study identified polymethyl methacrylate (PMMA) component degradation at elevated temperatures with isopropanol exposure. Recommendations include replacing PMMA with glass for improved durability. This work lays a foundation for implementing effective control strategies for the startup procedure of the closed-cycle distillation column. Once these strategies are in place, the system will be fully operational as a practical-based learning platform, effectively bridging the gap between academic theory and real-world chemical engineering practice.

1. Introduction

Distillation is a fundamental separation process widely used in the chemical industry to separate mixtures of liquids based on the differences in their volatility [1]. The distillation column investigated in this study is a 7-tray bubble cap column with a column diameter of 90 mm and transparent walls, providing clear visual access to the internal operations. Ambient pressure is approximately 87 kPa, but the pressure within the column will be slightly higher due to boil-up from the reboiler. Monitoring and control problems are the primary cause of column malfunctions [2], particularly during startup and shut down, therefore this is a critical component for safe and effective operation of distillation columns. Monitoring and control of the column were facilitated through the use of the NI DAQ hardware, in conjunction with a computer program called LabView.

At the start of this research, the operating window, defined as the range of operating conditions within which the column can function safely and efficiently, was unknown. Furthermore, there was no model of the system to describe its dynamic behaviour in response to its input variables, such as reflux flow, feed flow and reboiler duty. Without this model, developing and implementing a control strategy to facilitate the column's startup would be extremely challenging.

The FOPDT model, Equation 1, is known to adequately approximate the dynamic behaviour of most systems that have monotonic responses to step changes in input variables, [3]. Furthermore, FOPDT parameters can be used directly in tuning correlations, such as the Ciancone correlations found in [3], to obtain initial Proportional-Integral-Derivative (PID) tuning constants.

$$Y'(t) = K_p \Delta X \left[1 - e^{-\frac{t-\theta}{\tau}}\right] \tag{1}$$

$$Y'(t) = Y(t) - Y_{ss} \tag{2}$$

$Y'(t)$ denotes the deviation of the output variable from its initial steady-state value Y_{ss} at time t. ΔX is the step change in the input variable from its initial steady-state value. K_p approximates how the output variable changes in response to a change in the input variable, $\frac{\Delta Y}{\Delta X}$. θ approximates the process dead-time, which is the time taken for $Y'(t)$ to become non-zero following ΔX. τ describes the speed of the transient response of $Y'(t)$.

The Chemical Engineering Department intends to use the distillation system as a practical learning platform for its students. However, this requires a control strategy capable of facilitating startup and maintaining steady operation. Developing such a control system first requires a model of the system's dynamics, which can only be achieved through empirical data obtained from experiments designed

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to measure the dynamic behavior of the system in response to its inputs. This research, therefore, aims to derive and implement a control system that facilitates the startup of the distillation column, ultimately making it a functional platform for practical-based learning.

The objectives of this study were to first complete the physical system by finalising the setup of the distillation column and ensuring that all necessary hardware was installed and functional. The next step was to design and conduct experiments to obtain empirical data on the system's response to inputs such as reboiler duty and reflux flow. This data was then used to develop and fit FOPDT models that quantify the dynamic behavior of the system. Subsequently, the reliability and accuracy of these fitted models were analysed in the context of the column's operational behavior. Finally, the study aimed to implement PID control algorithms by deriving tuning constants from established literature, such as the Ciancone correlations.

The study completed the physical setup of the system and the NI DAQ was installed and configured. An attempt was made to experimentally obtain pump curves for the system's pumps, and various instruments were tested and verified to ensure they functioned within their intended ranges. Experiments to obtain empirical data capturing the system's temperature response to reboiler duty were performed, and FOPDT models were fitted to the data. An attempt to model the vapor-liquid equilibrium (VLE) behavior within the distillation column was made.

2. Experimental

2.1. Apparatus and materials

A simplified process flow diagram (PFD) of the system, Figure 1, depicts the layout of the internally custom-built distillation system and the specifics of the various components are included in Table 1.

A 70 % water—30 % isopropanol mixture was prepared using distilled water and 99.9 % propan-2-ol that was sourced from Promark Chemicals, Johannesburg, South Africa. This mixture was used for characterising the reboiler level and collecting empirical data for fitting FOPDT models. Municipal water was utilised in the experiments for obtaining the pump curve, verifying the manufacturer's turbine flowmeter specifications, characterising flow rates through control valves at different openness levels, and servicing utility streams in heat exchangers HX-01 and HX-02.

Table 1
Equipment of closed-cycle distillation column.

Equipment	Tag ID	Quantity	Description
Control valve	CV01-05	5	RCV pneumatic 1/2 inch
PDT[a]	PDT01-02	2	0 mm — 635 mm H_2O
Flowmeter	FT01-04	4	Turbine YF-S201
Pump	PMP01-03	3	Totton centrifugal PC40/6
Thermocouple	TT00-12	13	J-type
Reboiler	Reboiler	1	9 kW and 60 L
Condenser	HX01	1	Shell and tube
Heat exchanger	HX02	1	Double-pipe

[a] Pressure Differential Transmitter.

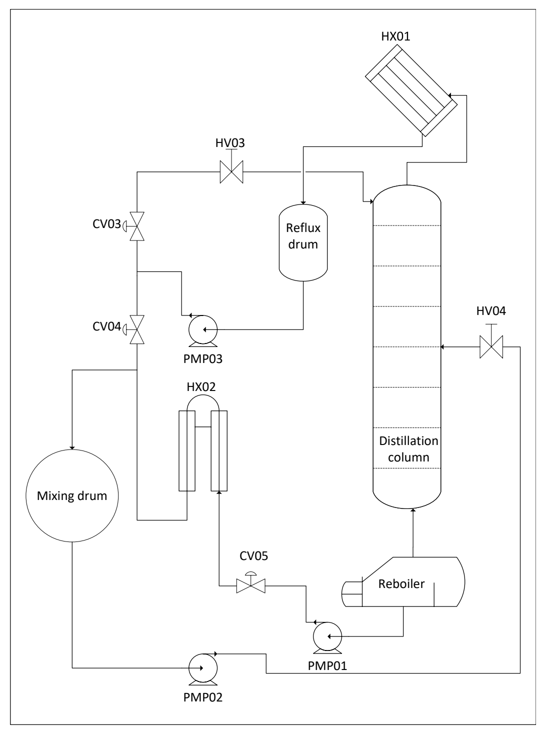

Figure 1: Process flow diagram of the system, showing only the piping, equipment, and instrumentation related to the process fluid. Piping and instrumentation associated with utilities have been excluded for simplicity.

2.2. Methods

2.2.1. System verification and calibration

The reboiler level was characterised by draining it and recording the signal from PDT01. Thereafter, increments of 5 L of the 70 % water—30 % isopropanol mixture were added to the reboiler and the signal from PDT01 recorded. Analysis of the increase in the signal between each interval is expected to identify a non-linear region, corresponding to the position of the heating elements. This marks the minimum reboiler level required to ensure that the heating elements are covered.

The experimentally determined pump curve data was obtained as follows: The suction side of the pump was connected to a water tank and the discharge side was connected to a 6 m long transparent silicon tube that was fed back into the water tank to close the loop. A Bürkert SE35 flowmeter was placed inline between the tube and the discharge side of the pump. By adjusting the tube's elevation above the pump, the water transport height was changed, and the corresponding signal from the flowmeter was recorded.

The turbine flowmeters were characterised on the system itself, using one of the flowmeters in the utility lines proceeding the tap that provides municipal water to the system. The discharge line of the flowmeter was opened into a bucket. The frequency signal of the flowmeter was recorded by the DAQ for several levels of openness of the tap. For each level of openness the flow was collected in the bucket, weighed and converted into a volumetric flow rate using the density[1] of water and the time over which the flow was collected.

The zero and span of the IP transducers were tested to verify that each transducer outputs 3 psi when a 4 mA signal is provided and 15 psi when a 20 mA signal is provided. The output of each transducer was measured by connecting its output to a Wika pressure transducer, which was wired to the DAQ system to display the measurement in psi. If required, the adjustment screws on the body of the transducer were used to correct the zero and then the span of the transducer.

Any brass pipe sections that were reconfigured during the process were reconnected with solder and leak tested, by running water through them and carefully observing to ensure that there were no leaks.

2.2.2. *Empirical data collection and model validation*

The startup procedure aimed to get a suitable vapor flow rate through the column and obtain a reasonable level in the reflux drum. Thereafter, reflux can be started and the distillate and bottoms were sent to the mixing drum and subsequently fed into the column. The procedure is an iterative method, that focussed on systematically bringing each aspect of the system into operation, while making the necessary adjustments to the overall system to achieve steady operation. The procedure was conducted with all the sensors of the system operating and the measurements recorded by the NI DAQ system.

From a cold start, all pumps were switched off and all control valves were closed. The reboiler contents were heated to the saturation temperature with the reboiler duty set to 60 % of its duty. At saturation the reboiler temperature was constant and the vapor flow rate through the column and the tray temperatures gradually began to increase. CV01 was opened when condensate was observed on the top tray to start the condenser. The reboiler duty was adjusted to produce a vapor flow rate sufficiently large to provide a suitable level in the reflux drum, while avoiding flooding on any of the trays and providing sufficient quantities of reflux and distillate. The procedure was halted at this stage, as reflux could not be introduced at low enough flow rates with CV03, resulting in the sudden cooling of all the trays and drainage of the reflux drum.

However, to enable lower flow rates of reflux and feed for future experiments, HV03 and HV04 were included as seen in Figure 1. The planned next steps would have involved initiating reflux by opening control valve CV03 with HV03 half open. The openness of CV03, HV03 and the reboiler duty would be adjusted to prevent excessive cooling or flooding in the column and maintain a stable level in the reflux drum. Before directing the distillate and bottoms to the mixing drum, the bottoms were to be cooled via HX02 to prevent cavitation in PMP02 and flashing in the mixing drum. Thus, the duty of HX02 would be set to ensure that the bottoms are cooled to a temperature below that of the distillate. Thereafter, the feed would be started and controlled using CV05, CV02 and HV04 such that no flooding or excessive cooling occurs within the column.

[1] $997\,\mathrm{kg/m^3}$

2.2.3. *Approximating system dynamics*

The typical experimental design, as described by [3], would be to introduce a step change in the reboiler duty and hold it constant until all transient behaviors of the temperature responses have ceased. In this study, due to flooding, the reboiler duty could not be held constant for the full duration, and adjustments were made before steady state was reached. FOPDT models were fitted using data from the initial step change, with Python's `SciPy` library function `scipy.optimize.curve_fit` to optimise K_p, θ and τ of Equation 1.

From a cold start, the temperature of the reboiler in a distillation column rises until it reaches the liquid's saturation temperature. At this point, boiling begins, and the temperature remains constant despite continued heat input, fundamentally changing the system and its behavior. This same process occurs on each tray within the column.

Consequently, the dynamic behavior of the system after reaching saturated conditions can not be estimated using models of the startup phase. The models fitted to the measured data during startup do not account for the system's proximity to saturation conditions. They also do not consider that, at saturation, the energy input no longer increases the sensible heat of the subcooled liquid but is instead consumed as the latent heat of vaporisation during the phase change. Therefore, separate models are necessary for the startup and operational phases to estimate the system's behavior.

The NRTL model can describe VLE behaviour of highly non-ideal systems at low pressures [4]. Thus, it can account for the significant non-ideal interactions, such as hydrogen bonding between water and isopropanol molecules. The NRTL thermodynamic property package, available in DWSIM, was used to estimate the saturation temperature of the 70 % water—30 % isopropanol mixture at 87 kPa. This value, along with the bubble point temperatures of pure water and pure isopropanol at 87 kPa, were used to establish a reference temperature range for determining whether a measured temperature corresponds to saturated conditions. In a distillation column, the temperatures of the trays vary along the column height: Trays nearer to the condenser tend toward the saturation temperature of the most volatile component (MVC), while trays closer to the reboiler approach the saturation temperature of the least volatile component (LVC) [5].

3. Results and Discussion

3.1. System verification and calibration

The minimum reboiler level ensuring coverage of the heating elements was identified at a PDT signal of 10 mA, corresponding to a volume of 20 L. As shown in Figure 2, the relationship between volume and pressure head in the reboiler appears linear after 10 L. The PDT, positioned above the reboiler base, likely only detects changes after this volume. A linear trend is expected along the bulk of the reboiler's profile, due to its uniform walls, with deviations in linearity in the region where the heating elements are inserted into its profile. This deviation was identified by analysing the first and second derivatives, estimated in Figure 3.

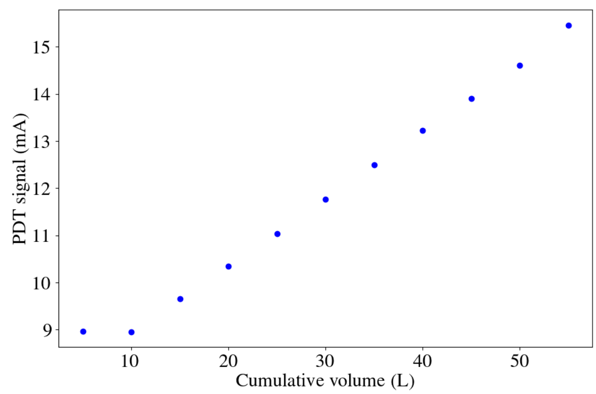

Figure 2: Signal of the PDT measuring the pressure head in the reboiler at each incremental increase of 5 L.

The first derivative should be constant until reaching the heating elements, then deviate, returning to its original constant value afterward, while the second derivative should be zero except in this region. The data largely supports this, though the first derivative remained zero until 15 L, likely due to the "Threshold Effect", where the PDT only detected changes at that point. While the data is not entirely conclusive due to the "Threshold Effect", where the PDT only began detecting changes around 15 L, the results are consistent with what can be estimated by inspecting where the elements are inserted into the reboiler's profile. Considering that the heating elements are positioned within the first third of the reboiler's height, it is reasonable to confirm their coverage at a PDT signal of 10 mA, as the 20 L mark corresponds to approximately one-third of the reboiler's volume.

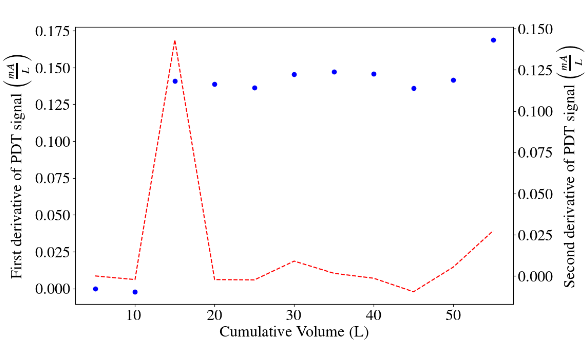

Figure 3: Dual-axis plot showing the first and second derivatives of the PDT signal with respect to cumulative volume. The blue dots represent the first derivative of the PDT signal (primary axis), while the red dashed line represents the second derivative of the PDT signal (secondary axis).

The experimentally determined pump curve, Figure 4, indicates that the flow rate is a constant at 20 L min^{-1} up to a pressure head of 3.5 m, where it is 0 L min^{-1}. These results are not useable as they do not align with the known behaviour for centrifugal pumps. The irregular results are likely due to the use of a flowmeter that is not suitable for the range of flow rates tested.

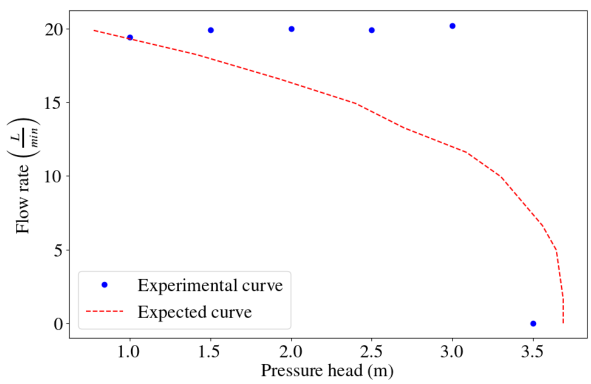

Figure 4: Comparison of the experimentally determined pump curve (blue dots) with a synthetically generated curve (red dashed line) representing the typical shape of a centrifugal pump curve.

The Empirically derived relationship for the turbine flowmeters is similar to that found in the manufacturer's data sheet, [6]. The fitted models are shown in Figure 5, where the gradients are 8.86 L h^{-1} Hz^{-1} and 7.78 L h^{-1} Hz^{-1} for the empirically determined model and the model fitted to the manufacturer's data, respectively.

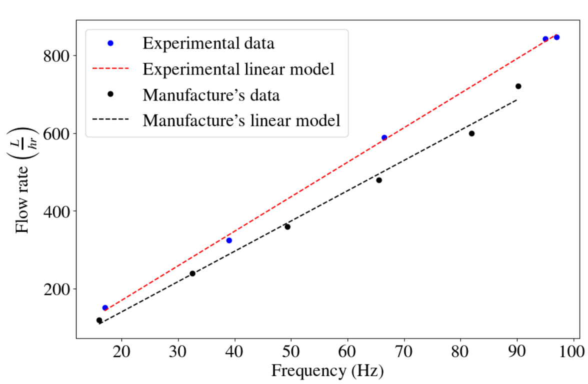

Figure 5: Plot comparing the empirically determined linear relationship to that obtained from the manufacturer's data sheet, for the YF-S201 turbine flowmeters.

3.2. FOPDT models

The following discusses the data collected from attempting the experimental procedure outlined in Section 2.2.2, and the FOPDT models that were fitted to the data. Before proceeding with data analysis, the experiments revealed that PMMA is incompatible with isopropanol at elevated temperatures. After the experiment, significant cracking was observed in the PMMA walls of the column, compromising

Table 2
Saturation temperatures at 87 kPa.

Component	Saturation Temperature (°C)
Pure Isopropanol	78.6
Pure Water	95.8
Mixture[a]	80.7

[a] 70% Water - 30% Isopropanol mixture.

the system's structural integrity. This degradation necessitated an immediate halt to further experimentation.

As anticipated, the reboiler duty could not be held constant until all transient responses had ceased, as depicted in Figure 6. The analysis is presented for the first 40 minutes of what was an 80 minute long experiment, because after 40 minutes the reboiler duty was changed so frequently that any analysis of the data thereafter would be highly speculative. Furthermore, tray 7 was excluded from the analysis due to concerns of the reliability of its measured data.

The temperature responses shown in Figure 6, each appear to plateau at temperatures within the range of the saturation temperature of isopropanol and the 70% water - 30% isopropanol mixture, shown in Table 2. Thus, for the analysis the plateau temperatures will be assumed to correspond to saturation temperatures. From Figure 6, the reboiler reaches its saturation temperature after approximately 21 minutes and the first 6 trays reach a steady state after approximately 38 minutes. Thus, the startup time for the first 6 trays with an average reboiler duty of 48.2 % is estimated as 38 minutes. Recall from Section 2.2.3 that different models are required

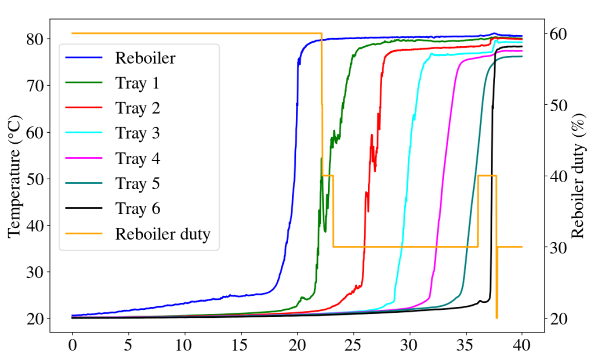

Figure 6: Dynamic temperature profiles of the reboiler and the first six trays are plotted on the primary y-axis, and the reboiler duty is plotted on the secondary y-axis. The reboiler duty is the system input and the temperature responses are the system outputs.

for predicting the system's dynamic response to step changes that occur before and after startup. All models and data presented in this section are for input step changes from a cold start when the reboiler is at ambient conditions of approximately 21 °C.

Provided that the reboiler duty is sufficiently high, the reboiler temperature is expected to remain at saturation

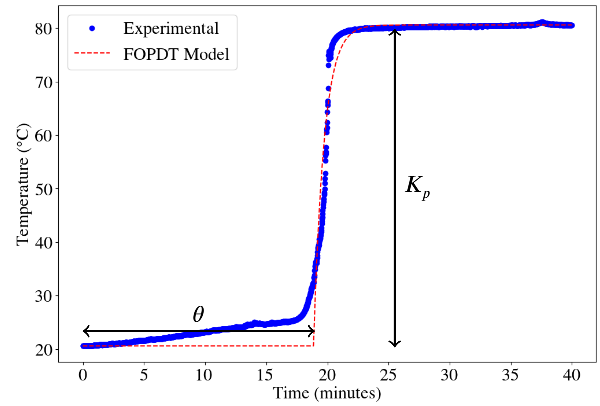

Figure 7: FOPDT model of the reboiler temperature response. $K_p = 1.00 \frac{°C}{\%\text{reboiler duty}}$, $\tau = 0.859\ \text{min}^{-1}$, and $\theta = 18.861$ minutes. This plot is annotated to depict how the FOPDT parameters K_p and θ mathematically contribute to the transient response of the reboiler. τ could not be conveniently annotated, but would be approximately the time that the system takes after θ minutes to reach roughly 63.3% of $\Delta X K_p$

during operation, gradually increasing toward the boiling point of water over time. Additionally the tray temperatures are expected to remain at their saturation temperatures given that the reboiler duty provides sufficient boil-up. This is consistent with the data depicted in Figure 6, where the reboiler temperature remains steady at approximately 80 °C and the tray temperatures plateau between 75 °C and 80 °C even after the reboiler is stepped down to 30 %.

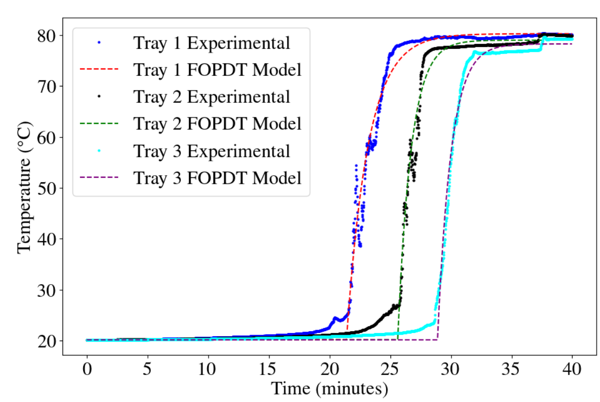

Figure 8: FOPDT models for trays 1, 2 and 3. The fitted parameters for the i^{th} tray are as follows: Tray 1: $K_{p1} = 1.00 \frac{°C}{\%\text{reboiler duty}}$, $\tau_1 = 1.776\ \text{min}^{-1}$, and $\theta_1 = 21.39$ minutes. Tray 2: $K_{p2} = 0.98 \frac{°C}{\%\text{reboiler duty}}$, $\tau_2 = 1.106\ \text{min}^{-1}$, and $\theta_2 = 25.612$ minutes. Tray 3: $K_{p3} = 0.968 \frac{°C}{\%\text{reboiler duty}}$, $\tau_3 = 1.117\ \text{min}^{-1}$, and $\theta_3 = 28.893$ minutes.

The FOPDT models were fitted using the initial reboiler step change, ΔX, of 60 % and the results indicate that the dynamic responses of the trays are quite similar. Excluding tray 6, the process gain (K_p) decreases from tray to tray moving up the column, as shown in Figure 10. This reduction in gain can be attributed to heat losses between trays, which increase with height, diminishing the proportion of energy from the reboiler that reaches each successive tray. However, as previously discussed the plateau temperatures of the trays resemble possible saturation temperatures for this system. In distillation systems, trays positioned higher in the column tend toward the boiling point of the more volatile component due to the increasing concentration of that component resulting from the separation process. Therefore, the apparent drop in K_p moving up the trays is likely a result of the separation process and the differing mixture compositions on each tray, rather than cumulative heat losses. Figure 11 shows that most

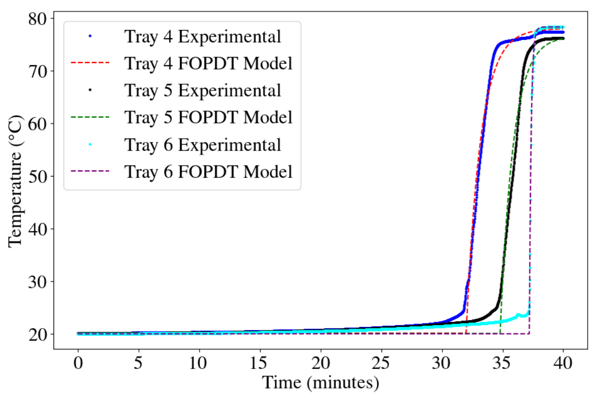

Figure 9: FOPDT models for trays 4, 5 and 6. The fitted parameters for the i^{th} tray are as follows: Tray 4: $K_{p4} = 0.964 \frac{°\mathrm{C}}{\%\mathrm{reboiler\ duty}}$, $\tau_4 = 1.187$ min^{-1}, and $\theta_4 = 31.995$ minutes. Tray 5: $K_{p5} = 0.941 \frac{°\mathrm{C}}{\%\mathrm{reboiler\ duty}}$, $\tau_5 = 1.079$ min^{-1}, and $\theta_5 = 34.795$ minutes. Tray 6: $K_{p6} = 0.971 \frac{°\mathrm{C}}{\%\mathrm{reboiler\ duty}}$, $\tau_6 = 0.141$ min^{-1}, and $\theta_6 = 37.207$ minutes.

trays exhibit similar time constants, τ, indicating comparable speeds in their transient responses. Notably, stages 1 and 6 deviate, with stage 1 responding significantly slower than stages 2 to 6. Recall that τ reflects the system's speed post-dead-time. For stage 1, the dead-time was 21.39 minutes, which marked the start of its transient response. Between 22.2 and 23.2 minutes, the reboiler duty was stepped down to 30 %, interrupting and slowing tray 1's transient response before it could reach completion. This effect is evident in Figure 8, where irregularities in stage 1's data points are observed around 23 minutes. Subsequent trays' time constants were unaffected, as their larger dead-times prevented their transients from starting during the reboiler duty change, thus, likely increasing their θ values.

By observing the response of tray 1 to the reboiler duty step at 22.2 minutes in Figure 6, it's evident that once boil-up begins, there is no observable dead-time between changes in the reboiler duty and the temperature response of tray 1. Thus, the dynamic response of tray 1 to changes in reboiler duty post reboiler saturation are much faster.

As seen in Figure 12, dead-time increases moving up the column. Figure 13 represents the approximate dead-time between adjacent stages in the system during startup. The sharp increase in $\Delta\theta$ between stage 1 and stage 2 can be attributed to the slowed transient response of tray 1,

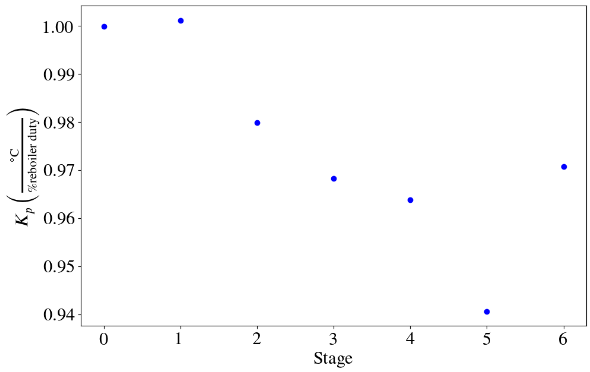

Figure 10: Plot of K_p of the FOPDT model, for stages 0 to 6 where, stage 0 is the reboiler and stage 6 is the sixth tray of the distillation column.

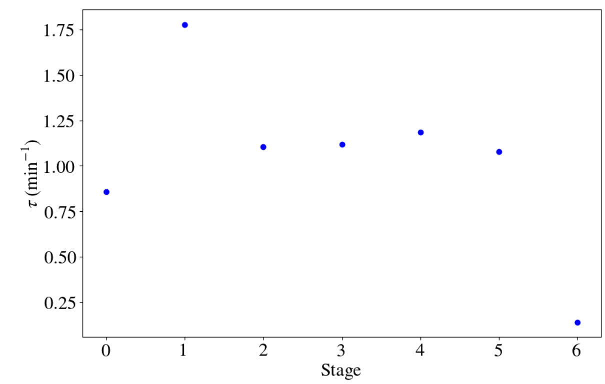

Figure 11: Plot of τ of the FOPDT model, for stages 0 to 6 where, stage 0 is the reboiler and stage 6 is the sixth tray of the distillation column.

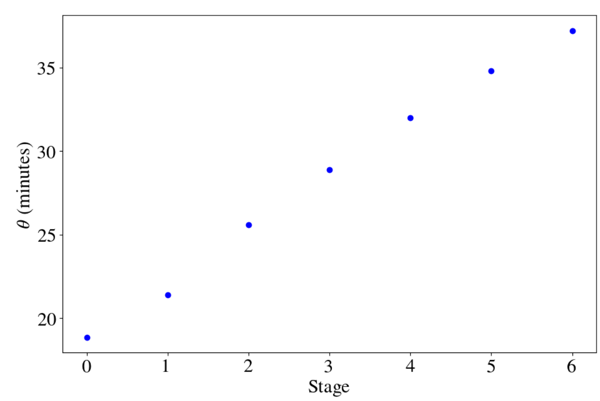

Figure 12: Plot of θ of the FOPDT model, for stages 0 to 6 where, stage 0 is the reboiler and stage 6 is the sixth tray of the distillation column.

caused by the step-change in reboiler duty at 22.2 minutes, as discussed earlier. Additionally, $\Delta\theta$ decreases moving up the column, suggesting shorter delays between stages.

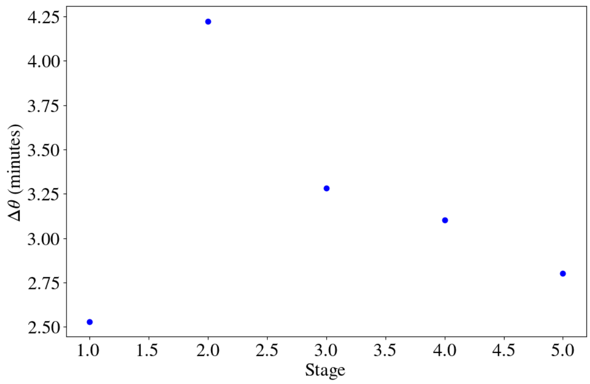

Figure 13: Plot of $\Delta\theta$ at each stage, where $\Delta\theta = \theta_i - \theta_{i-1}$, representing the difference between θ at the current stage and the previous stage, for stages 1 to 6. This is essentially the θ between adjacent trays during startup.

4. Conclusions

The physical setup was successfully completed with all necessary hardware installed, tested, and configured, including the NI DAQ system. Experiments were then designed and conducted, providing empirical data on the temperature response of the system to changes in the reboiler duty during startup.

The first six trays and the reboiler were modeled using FOPDT models, with tray 7 being excluded from this study due to concerns over the reliability of its measured data. The FOPDT models revealed that, apart from tray 6, the tray responses were generally similar with slight variations in process gains and time constants. It was found that the reboiler reaches saturated conditions after approximately 21 minutes with its duty set to 60 %, and the the first six trays reach saturated conditions after approximately 38 minutes with an average reboiler duty of 48.2 %. Thus, the estimated startup time for the first 6 trays with an average reboiler duty of 48.2 % is 38 minutes.

The analysis of these models, in relation to the saturation temperatures of pure isopropanol and the 70 % water—30 % isopropanol mixture, suggests that at a reboiler duty of 30 % or higher, the steady-state temperatures are governed by the saturation temperature of the respective liquid mixture rather than by heat losses. Thus, the process gain (K_p) for each model is a function of the mixture composition on the respective tray.

Empirical data where reflux and feed were introduced could not be included, as the introduction of reflux caused significant cooling across the system. It was determined that the heating elements in the reboiler are fully submerged when the signal from PDT01 reaches 10 mA. Additionally, in the absence of reflux and feed, the system can maintain saturated conditions with a reboiler duty as low as 30 %. However, flooding is likely to occur at reboiler duties of 60 % or higher.

For future work, it is recommended that the thermocouple on tray 7 be tested to verify its functionality. Additionally, all PMMA sections of the column and the condenser should be replaced with glass.

To gain a more comprehensive understanding of the system's operational limits, experiments should be conducted to determine the minimum reboiler duty necessary to maintain all trays at saturated conditions. This will help establish a lower bound for the reboiler's operational window, ensuring that trays do not begin to subcool during steady operation. The experiment to determine the pump curve should be repeated using a more suitable flowmeter.

Additionally, the startup procedure should be repeated, incorporating reflux and feed flows into the system as outlined in Section 2.2.2. These experiments will provide the empirical data required to model the system's dynamic behaviour with reflux and feed. Thereafter, control strategies to facilitate the startup of the distillation system can be developed using the FOPDT models obtained in this study, and those to be developed for reflux and feed.

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