Optimal Energy Consumption of the Distillation Process and Its Product Purity Analysis Using Ultraviolet Spectroscopy

Eadala Sarath Yadav, Thirunavukkarasu Indiran,* and S. Shanmuga Priya

ABSTRACT: This paper addresses the energy consumption of distillation process via an actuator, which is a challenging problem in process industries. Precise control action would enhance energy consumption and improve the productivity. This paper is an experimental validation of EPC-PI control algorithm and analysis of distillate purity of a lab-scale distillation column. The PI control scheme uses closed-loop data of extended predictive controller (EPC) that has been performed through off-line simulation. The performance of control method is compared with different schemes such as Hägglund’s one-third rule and Skogestad’s overshoot method. The issue of integral windup in the multivariable process is addressed in the aspect of optimal energy consumption. The energy consumption calculations are made with respect to power utility of actuators throughout the process. The distillate product of post-controller implementation is processed to qualitative analysis using UV spectroscopy. Performance index is carried out via integral time absolute error (ITAE) by perturbing plant parameters up to 30% uncertainty.

1. INTRODUCTION

The growth of process industries such as pharmaceutical, chemical, automobile, and food processing is highly progressing due to their demand in day-to-day life. These industries have been contributing toward the growth of the country’s economy as well. According to a study by the Associated Chambers of Commerce and Industry of India (ASSOCHAM), the petrochemical industry in India would reach 100 billion dollars by 2020. In view of extensive growth of process industries, energy utilization for their operations has become significant. According to the U.S energy information administration, the industrial sector uses 54% of world’s total delivered energy. The chemical industry is one such area that requires much attention in the aspect energy consumption due to its demand. Precise operation of chemical processes helps in achieving maximum productivity and improve energy consumption. Focus on mathematical modeling and controller design makes plant operations simple and more effective. There are quite a good number of literature available on methods of mathematical modeling. Sujatha and Panda presented a brief idea of interaction of loops in the MIMO process and how to overcome this problem by using the control configuration selection among the loops. They also implemented a sequential relay test for the parameter estimation MIMO process. Although there exist many papers on estimating the plant model in terms of FOPDT, most of the papers neglect the estimation of dead time. Luyben worked on the identification of FOPDT and SOPDT models by using an autotuned relay method only when the steady-state gain was known a priori.

Control design methods are broadly categorized into three types, PID control, traditional controller with advanced loops such as cascade, feedforward, etc., and model predictive control (MPC). Although there exist different control schemes, most of the methods developed in the literature are limited to the simulation environment. Practical issues of industrial processes such as integral windup, derivative kick, bump-less transfer, and multivariable interaction could be apparently realizable in real-time implementation. The efficient control scheme can handle the process terms such as time delay, system constraints (soft and hard), and model mismatch. The distillation process is one of the chemical systems that exhibit complex dynamics and it is difficult to control because of high interaction between the input and output variables. Maghade and Patre had designed PI/PID controllers for the two-input two-output (TITO) process using gain and phase margin specifications. Le Gorrec et al. presented the modeling, identification, and compensation of...
hysteresis in multi-degree of freedom in the actuated systems. Cornieles et al.\textsuperscript{14} gave a comparative survey on different multivariable control techniques in the field process control.

The fundamental objective of this work is to minimize the energy consumption of the actuators in the closed-loop control applications. The attempt has been made to address the practical issue of integral windup for the MIMO process. The proposed EPC-PI has been implemented in the experimental environment and has validated the efficiency of the method through controller responses; therefore, energy consumption of the actuator is estimated. In addition, the distillate product purity has been analyzed from the initial feed composition. This paper is organized as follows: Section 2 provides detailed view of mathematical models considered in this work along with different control schemes including the proposed EPC-PI controller. The simulation and experimental results of different control schemes are presented in Section 3. Section 4 deals with the analytical study of distillate purity using UV spectroscopy. The last section is dedicated to conclusions.

2. MATHEMATICAL MODELS AND CONTROL SCHEMES

2.1. Mathematical Model. The mathematical modeling plays a significant role in controlling any process. The control over a process is efficient if the model identification is optimal. The purpose of identifying the model is to represent the physical system in terms of mathematical equations to design an optimal controller to control the process. The black-box mathematical modeling approach is considered in the work. As the plant is nonlinear, the system is linearized around an operating region. The input–output data obtained from a lab-scale batch distillation column at a particular operating region is subjected to a linear regression modeling method.\textsuperscript{15} The schematic diagram of the experimental setup considered in this work is shown in Figure 1.

The model structure is considered as FOPDT and the parameters are obtained as follows

Note that the detailed methodology and experimental responses related to the model (eq 1) is presented in the preceding version of this work.\textsuperscript{16}

The effective transfer function models using the methodology presented by Yadav et al.\textsuperscript{16} are shown in eqs 2 and 3.

\begin{equation}
\hat{G}(s) = \frac{Y_1(\text{tray}-5)}{X_1} = \begin{bmatrix}
-0.1406s^{-0.0142} \\
-0.3929s^{-0.166} \\
0.083e^{-0.011s} \\
1.04s + 1 \\
0.0142 \\
0.012 \\
0.5985s + 1 \\
0.6335s + 1 \\
0.164s + 0.006s^{-0.006s} \\
0.3191s + 1 \\
\end{bmatrix}
\end{equation}

\begin{equation}
G_{11\text{CL}}(s) = \frac{-0.417e^{-0.082s}}{1.7s + 1}
\end{equation}

\begin{equation}
G_{22\text{CL}}(s) = \frac{0.083e^{-0.11s}}{0.32s + 1}
\end{equation}

2.1.1. Wood and Berry Model. The Wood and Berry model\textsuperscript{18} is considered a benchmark model for the MIMO process. It has been identified by exciting the reflux flow and steam flow down the column, by which they cause the change in the overhead and the bottom compositions in a binary distillation column. It is proven to be a difficult process to control because of an interaction effect between the input and output variables.
The variable of interest is the temperature at the 17th and 24th trays of the distillation column for separating methanol and water. The models for the MIMO process are obtained as effective transfer function models given in Eqs 5 and 6:

\[ G(s) = \begin{bmatrix} 12.8e^{-1s} & -18.6e^{-3s} \\ 16.7s + 1 & 21s + 1 \\ 6.6e^{-7s} & -19.4e^{-3s} \\ 10.9s + 1 & 14.4s + 1 \end{bmatrix} \] (4)

The FOPDT structure of the Wood and Berry model (Eq 4) is used to implement the EPC-PI control scheme. It is the study of 24 tray-distillation column model\(^\text{19}\) tends to be one of the benchmark processes. This method is similar to the Ziegler–Nichols open-loop tuning method,\(^\text{22}\) but the controller gain is set to one-third of the Ziegler–Nichols controller value. Therefore, the closed-loop response would not attain sustained oscillations but overshoot; indeed, the overshoot is a criterion to design the controller parameters. The relationship is obtained from the closed-loop step input response, where a proportional controller is recommended as a function of the first peak, i.e., overshoot, and the integral time is considered a function of peak time of the process as shown in Eqs 11 and 12. There is also a detuning factor incorporated within the controller parameters to adjust the gains such that the user gets the desired stability and robustness.

Controller parameters are obtained as:

- **Proportional gain (Kp):**
  \[ K_p = \frac{K_p A}{F} \] (11)

where Kp is the proportional gain used to obtain the required overshoot A = [1.152(overshoot)\(^2\) − 1.607(overshoot) + 1.0].

- **Integral time (Ti):**
  \[ T_i = \min \left( \frac{0.86A}{1 - b}, \frac{t_P}{2.44t_fF} \right) \] (12)

The detuning factor needs to be selected as F > 1 for more robustness and, for fast response, F should be selected as F < 1. The b is obtained from the closed-loop response from Figure 3 as \( b = \frac{\Delta y_1}{\Delta y_2} \) or \( b = \frac{KK_p}{(1 + KK_p)} \) and \( t_p \) is the time for the process to reach its overshoot or the first peak.

**2.4. EPC-PI Method.** The controller parameters of the proposed method presented in our preceding work is given in Eqs 13 and 14.

- **Proportional gain:**
  \[ K_p = \frac{1}{3} \] (13)

where the tuning parameter \( \lambda \) is selected as the minimum value of diagonal time constant for loop-2 and the maximum value of off-diagonal time constant for loop-1.

\[ \lambda_1 = \max \{r(G_{12}, G_{22}) \} \]
\[ \lambda_2 = \min \{r(G_{11}, G_{22}) \} \]
Ts is the settling time of EPC closed-loop response and Δu is the change in controller output.

Integral gain:

\[ K_I = \frac{K_P}{T\alpha} \]  

(14)

where the rise time of EPC response is selected as Tr and the speed of the process is influenced by using the tuning factor \( \alpha \) with the range of (0,1].

Note that the detailed methodology of EPC-PI control is presented in the preceding version of this paper.16

3. RESULTS AND DISCUSSION

Three models, namely, the Wood and Berry model (eq 4), Vinante and Luyben model (eq 7), and experimental model (eq 1), were approximated into effective transfer function models through the methodology given in ref 17. Three different PI/PID control schemes (Hägglund’s one-third rule, Skogestad’s overshoot method, and proposed scheme) have been designed and simulated and the experimental model has been validated on a lab-scale distillation column. The results are as follows.

3.1. Case Study-1: Wood and Berry Model. The Wood and Berry model (eq 4) is one of the benchmark model for the MIMO process and has been identified by exciting reflux flow and steam flow down the column by which they cause the change in the overhead and bottom compositions in a binary distillation column. It is proven to be a difficult process to
control because of an interaction effect between the input and output variables.

3.1.1. Servo Operation. The servo operation with step changes of \([1,2]\) is applied at sampling time instants of \((0,200)\) min for loop-1. The corresponding response of \(Y_1\) is observed in Figure 4 and the corresponding manipulated variable response is depicted in Figure 5.

For loop-2, the response of process variable \(Y_2\) with setpoint step changes of \([2,4]\) applied at time \((0,200)\) min is represented.
3.1.2. Regulatory Operation. Regulatory operation is performed by imposing load individually on both the loops at the initial state of the process. Figure 8a shows the response of $Y_1$ when the load is applied on $Y_1$ and Figure 8b depicts the response of $Y_2$. Similarly, Figure 9a shows the response of $Y_1$ with the load on $Y_2$ and Figure 9b depicts the response of $Y_2$ with the load on $Y_2$.

### Figure 14. Load operation of $Y_1$ and $Y_2$ with the load on $Y_1$ for the VL model.

### Figure 15. Load operation of $Y_1$ and $Y_2$ with the load on $Y_2$ for the VL model.

3.2. Case Study-2: Vinante and Luyben Model.

3.2.1. Servo Operation. The Vinante and Luyben distillation column model (eq 7) is the study of separating methanol and water. The procedure of implementation is the same as the Wood and Berry model. Figures 10 and 11 depict the responses of $Y_1$. Similarly, Figures 12 and 13 show the responses of $Y_2$.

### Figure 16. Servo response comparison of $Y_1$ for the lab-scale model.

### Figure 17. Manipulated variable response comparison of loop-1 for the lab-scale model.

### Figure 18. Servo response comparison of $Y_2$ for the lab-scale model.

### Figure 19. Manipulated variable response comparison of loop-2 for the lab-scale model.

in Figure 6 and the corresponding manipulated variable is given in Figure 7.

3.1.2. Regulatory Operation. Regulatory operation is performed by imposing load individually on both the loops at the initial state of the process. Figure 8a shows the response of $Y_1$ when the load is applied on $Y_1$ and Figure 8b depicts the response of $Y_2$. Similarly, Figure 9a shows the response of $Y_1$ with the load on $Y_2$ and Figure 9b depicts the response of $Y_2$ with the load on $Y_2$.
of $Y_2$ is depicted in Figure 14b. Similarly, Figure 15a shows the response of $Y_1$ with the load on $Y_2$. For the same load on $Y_2$, the response of $Y_2$ is depicted in Figure 15b.

3.3. Case Study-3: Lab-Scale Distillation Column Model. 3.3.1. Servo Operation. The lab-scale distillation column model represented in eq 1 is subjected to the servo operation with step changes of [1,2] applied at sampling time instants of (0,200) min for loop-1. The corresponding response of $Y_1$ is observed in Figure 16 and the corresponding manipulated variable is given in Figure 17. For loop-2, the response of process variable $Y_2$ with servo operation of [2,4] applied at time (0,200) min is represented in Figure 18 and the corresponding manipulated variable is given in Figure 19.

3.3.2. Regulatory Operation. Regulatory operation for eq 1 is simulated as the same methodology followed in Section 3.1 Figure 20a depicts the response of $Y_1$ when load is applied on $Y_1$. For the same load on $Y_1$, the response of $Y_2$ is shown in Figure 20b.

Similarly, Figure 21a depicts the response of $Y_1$ when the load is applied on $Y_2$. For the same load on $Y_2$, the response of $Y_2$ is shown in Figure 21b.

3.4. Experimental Implementation of Different Control Algorithms. Energy consumption of any system depends on different variables involved in it. In industries, an actuator is one such variable that consumes more energy. The batch process encounters quite different uncertainties in their
operations. In the presence of uncertainties, the process output may get deviated; in such cases, the control input must be optimal to withstand the process output from the undesired deviations. The appropriate modeling and control methods help overcome those uncertainties, which in turn results in a cost-effective solution. One of major problems in experimental validation is integral windup. It is a phenomenon that occurs when the controller output exceeds the saturation limit of the actuator. Anti-reset windup is a method of adding an additional

![Figure 23. Closed-loop response of the setpoint overshoot method on the experimental system.](image)

![Figure 24. Closed-loop response of the setpoint proposed method on the experimental system.](image)

| Table 1. Energy Consumption of Different Controllers |
|----------------------------------------------------|
| control scheme | energy consumption (Wh) |
|----------------|-------------------------|
| EPC-PI (proposed) | 2908 |
| one-third rule | 3550 |
| setpoint overshoot | 4024 |

| Table 2. Comparison of ITAE with Uncertainty (Servo Operation) |
|---------------------------------------------------------------|
| method | main | interaction | total |
|--------|------|-------------|-------|
| C-1    | Hägglund (2019) | 229.34 | 234.19 | 463.53 |
| Skogestad (2010) | 48.18 | 45.8 | 93.98 |
| proposed | 53.56 | 46.14 | 99.7 |
| C-2    | Hägglund (2019) | 265.9 | 276.85 | 542.75 |
| Skogestad (2010) | 53.95 | 53.16 | 107.11 |
| proposed | 54.92 | 48.54 | 103.46 |
| C-3    | Hägglund (2019) | 259.2 | 270.27 | 529.47 |
| Skogestad (2010) | 50.81 | 50.04 | 100.85 |
| proposed | 59.32 | 62.13 | 121.45 |
| C-4    | Hägglund (2019) | 260.1 | 271.15 | 531.25 |
| Skogestad (2010) | 51.23 | 50.48 | 101.71 |
| proposed | 51.37 | 45.98 | 97.35 |
Table 3. Comparison of ITAE with Uncertainty (Load Operation)

| method | main | interaction | total |
|--------|------|-------------|-------|
| C-1    | 246.77 | 248.73 | 495.50 |
| Skogestad (2010) proposed | 55.56 | 52.04 | 107.60 |
| C-2    | 284.60 | 293.80 | 578.40 |
| Skogestad (2010) proposed | 65.42 | 56.45 | 121.87 |
| C-3    | 277.80 | 287.20 | 565.00 |
| Skogestad (2010) proposed | 58.61 | 57.07 | 115.68 |
| C-4    | 278.80 | 288.10 | 566.90 |
| Skogestad (2010) proposed | 59.04 | 57.48 | 116.52 |
| proposed | 52.61 | 44.72 | 97.33 |

In this work, a one-time feed charge of 10 L with 20% isopropyl alcohol (IPA) and 80% water is considered for the experimentation. Once the feed is charged with the batch distillation, any control algorithm needs experimentation. Once the feed is charged with the batch distillation, any control algorithm needs experimentation. Once the feed is charged with the batch distillation, any control algorithm needs experimentation. Once the feed is charged with the batch distillation, any control algorithm needs experimentation. Once the feed is charged with the batch distillation, any control algorithm needs experimentation.

The spectroscopy equipment consists of a light source, monochrometer, beam splitter, sample compartment, and detector. Therefore, it is necessary to consider the energy consumption of the actuator to validate the efficiency of the control schemes.

3.5. Energy Consumption Calculations. The two variables that influence the energy consumption in this work are as follows:

1. Heater wattage (2000 W)
2. Peristaltic pump wattage (22 W)

The saturation limit of heater voltage is limited to 0.80% and isopropyl alcohol (IPA) and 80% water is considered for the experimentation. Once the feed is charged with the batch distillation, any control algorithm needs experimentation. Once the feed is charged with the batch distillation, any control algorithm needs experimentation. Once the feed is charged with the batch distillation, any control algorithm needs experimentation. Once the feed is charged with the batch distillation, any control algorithm needs experimentation. Once the feed is charged with the batch distillation, any control algorithm needs experimentation.

As a control engineer’s prospective, the fundamental task is to maintain the temperature of trays to obtain the maximum purity in the column. However, it is also important to analyze the product purity by the measure of product’s concentration. This analysis can be performed by using analytical instruments such as UV spectroscopy, liquid chromatography, etc. UV spectroscopy is the setup considered for this work. The spectroscopy equipment is one of the machines used where chemical components are analyzed using the principle of ultraviolet—visible absorption. A detailed theoretical background is presented in the doctoral thesis. Isopropyl alcohol and water are selected as the test components. The feed of distillation is 20% isopropyl alcohol and 80% water. The boiling point of isopropyl alcohol is 82.5 °C. Different control algorithms have been implemented in the experimental setup. The product purity has been tested post implementation of the proposed control algorithm.

4. PURITY ANALYSIS

As a control engineer’s prospective, the fundamental task is to maintain the temperature of trays to obtain the maximum purity in the column. However, it is also important to analyze the product purity by the measure of product’s concentration. This analysis can be performed by using analytical instruments such as UV spectroscopy, liquid chromatography, etc. UV spectroscopy is the setup considered for this work. The spectroscopy equipment is one of the machines used where chemical components are analyzed using the principle of ultraviolet—visible absorption. A detailed theoretical background is presented in the doctoral thesis. Isopropyl alcohol and water are selected as the test components. The feed of distillation is 20% isopropyl alcohol and 80% water. The boiling point of isopropyl alcohol is 82.5 °C. Different control algorithms have been implemented in the experimental setup. The product purity has been tested post implementation of the proposed control algorithm.

4.1. Working of UV Spectroscopy. UV spectroscopy is fundamentally used for concentration analysis of chemical components. It basically determines the concentration in terms of absorption in the range of UV wavelength (10–400 nm). The spectroscopy equipment consists of a light source, monochrometer, beam splitter, sample compartment, and detector as shown in Figure 25.
When light is emitted on the monochromator, the beam of light splits into different lanes by making use of a prism. The refracted beam is sent through the sample compartment where the sample concentration is compared with a reference cell, which is also known as blank. Water is used as the reference cell because the entire beam of light would pass through it since no molecule would absorb the light. The detector is used to measure the intensity of light coming from sample compartments. The absorption of light of sample cell is calculated using the intensity difference \( I_0 - I \). The experimental setup of UV spectroscopy equipment is given in Figure 26. This spectroscopy equipment is installed with multifunctional control software called UV Probe by Shimadzu. It has the facility of capturing the intensity of a component in terms of absorption at different wavelengths.

4.1.1. Testing and Validation. The experiment is conducted for three different samples with reference to water. The samples are pure isopropyl alcohol, feed (20% isopropyl alcohol and 80% water), and distillate (top product). Initially, a blank test is performed on a UV spectroscopy equipment by inducing water inside both the sample compartments. Once the system is initialized, the sample test can be performed using different samples. Figure 27 depicts the spectrum of different samples in terms of absorption vs wavelength.

The product purity is estimated using the peak absorption of each sample. At a wavelength of 195 nm, it is observed that the maximum absorption of pure isopropyl alcohol is 3.7 abs. In reference to the maximum absorption of pure isopropyl alcohol, the distillate is obtained as 2.9 abs and the feed is observed to be 1.8 abs. Therefore, through the spectrum, it appears that approximately 60% purity is obtained from feed components.
The product is also tested using the fixed-wavelength UV spectroscopy. The wavelength of UV spectroscopy is kept at 210 nm and the absorbance of the product is observed as shown in Table 4. It is observed that 68% purity has been obtained using the fixed-wavelength UV spectroscopy.

5. CONCLUSIONS

This work attempts to validate the energy consumption of lab-scale stillillation process with respect to actuator power utility through different control schemes. The proposed EPC-PI has been compared with different control schemes such as the setpoint overshoot method\(^\text{(14)}\) and one-third rule.\(^\text{(20)}\) The control schemes are tested in the simulation environment for three different distillation plant models and implemented on a pilot plant experimental setup. From the energy calculations (refer to Section 3.5), it is observed that the proposed EPC-PI shows 38% and 22% improvement in energy consumption compared to setpoint overshoot and one-third rule correspondingly. The product composition obtained after successful implementation of the control scheme on the experimental setup is processed to analyze the efficiency of the purity through UV spectroscopy. Two types of UV spectroscopy systems are used to test the purity of the composition. It appears that approximately 60 and 68% purity values have been obtained using the variable and fixed-wavelength UV spectroscopy correspondingly with respect to the feed concentration of isopropyl alcohol.

### AUTHOR INFORMATION

**Corresponding Author**

Thirunavukkarasu Indiran — Department of Instrumentation and Control Engineering, Manipal Institute of Technology, Manipal Academy of Higher Education, Manipal 576104, India; orcid.org/0000-0001-7157-5395

**Authors**

Eadala Satish Yadav — Department of Instrumentation and Control Engineering, Manipal Institute of Technology, Manipal Academy of Higher Education, Manipal 576104, India; orcid.org/0000-0002-1829-7362

S. Shanmuga Priya — Department of Chemical Engineering, Manipal Institute of Technology, Manipal Academy of Higher Education, Manipal 576104, India

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.0c05731

**Notes**

The authors declare no competing financial interest.

### ACKNOWLEDGMENTS

The first author would like to acknowledge Manipal Institute of Technology, Manipal Academy of Higher Education, for providing scholarship under TMA Pai scholarship toward this experimental research work. The authors would like to acknowledge Dr. J. Prakash, Anna University, Tamilnadu, India, and Dr. Shreesha C, Professor and Head, Intrumentation Control Engineering, Manipal Institute of Technology, Manipal, for their continuous guidance in this work.

### NOMENCLATURE

- \(\vartheta\): dead time
- C-1: nominal plant
- C-2: uncertainty in \(K\)
- C-3: uncertainty in \(K\) and \(T\)
- C-4: uncertainty in \(K\), \(T\), and \(\vartheta\)
- EPC: extended predictive control
- \(F\): detuning factor
- FOPDT: first order plus dead time
- GPC: generalized predictive control
- IO: input—output
- ITAE: integral time absolute error
- \(K_p\): process gain
- \(L_r\): reflux flow ratio
- \(m\): control horizon
- MIMO: multi-input multi-output
- MPC: model predictive control
- \(n\): total run time
- \(p\): prediction horizon
- PID: proportional integral and derivative
- \(Q_h\): heater voltage
- SISO: single-input single-output
- SOPDT: second order plus dead time
- \(T\): time constant
- \(T_e\): elapsed time
- \(T_s\): sampling interval
- TITO: two-input two-output
- UV: ultraviolet
- VL: Vinante and Luyben
- WB: Wood and Berry
- Wh: watt hour

### REFERENCES

1. Siripuram, R. Indian petrochemical industry to reach 100bn dollars by 2020, says study; 2015; https://www.chemicals-technology.com/uncategorised/newsindian-petrochemical-industry-reach-100bn-by-2020-study-4562621/.

2. Administration, U. E. I. Industrial sector energy consumption; https://www.eia.gov/outlooks/ieo/pdf/industrial.pdf.

3. Edgar, T. F.; Himmelblau, D. M.; Lasdon, L. S. Optimization of chemical processes; McGraw-Hill, 2001.

4. Sujatha, V.; Panda, R. C. Control configuration selection for multi input multi output processes. *J. Process Control* 2013, 23, 1567–1574.

5. Panda, R. C.; Vijayaraghavan, S. Parameter estimation of linear MIMO systems using sequential relay feedback test. *AIChE J.* 2014, 60, 1672–1681.

6. Majhi, S. Relay based identification of processes with time delay. *J. Process Control* 2007, 17, 93–101.

7. Luyben, W. L. Derivation of transfer functions for highly nonlinear distillation columns. *Ind. Eng. Chem. Res.* 1987, 26, 2490–2495.

8. Ogawa, M.; Kano, M. Practice and challenges in chemical process control applications in Japan. *IFAC Proc. Vol.* 2008, 41, 10608–10613.

9. Vilanova, R.; Visioli, A. PID control in the third millennium; Springer: 2012, DOI: 10.1007/978-1-4471-2425-2.

10. Morari, M.; Gentilini, A. Challenges and opportunities in process control: Biomedical processes. *AIChE J.* 2001, 47, 2140–2143.

11. Muddu, M.; Narag, A.; Patwardhan, S. C. Reparametrized ARX models for predictive control of staged and packed bed distillation columns. *Control Eng. Pract.* 2010, 18, 114–130.

12. Maghade, D. K.; Patre, B. M. Decentralized PI/PID controllers based on gain and phase margin specifications for TITO processes. *ISA Trans.* 2012, 51, 550–558.

13. Habineza, D.; Rakotondrabe, M.; Le Gorrec, Y. Bouc-Wen modeling and feedback control of multivariable hysteresis in piezoelectric systems: Application to a 3-DoF piezotube scanner. *IEEE Trans. Control Syst. Technol.* 2015, 23, 1797–1806.

14. Cornieses, E.; Saad, M.; Gauthier, G.; Salah-Hassane, H. Modeling and simulation of a multivariable process control. In 2006...
IEEE International Symposium on Industrial Electronics; 2006; pp. 2700–2705.

(15) Fedele, G. A new method to estimate a first-order plus time delay model from step response. J. Franklin Inst. 2009, 346, 1–9.

(16) Yadav, E. S.; Indiran, T.; Priya, S. S.; Fedele, G. Parameter Estimation and an Extended Predictive-Based Tuning Method for a Lab-Scale Distillation Column. ACS Omega 2019, 4, 21230–21241.

(17) Sarath, E.; Indiran, T.; et al. Parameter Estimation and Control of a Pilot Plant Binary Distillation. J. Adv. Res. Dyn. Control Syst. 2017, 877.

(18) Wood, R. K.; Berry, M. W. Terminal composition control of a binary distillation column. Chem. Eng. Sci. 1973, 28, 1707–1717.

(19) Luyben, W. L.; Vinante, C. D. Experimental studies of distillation decoupling. Kem. Teollisuus 1972, 29, 499–514.

(20) Hågglund, T. The one-third rule for PI controller tuning. Comput. Chem. Eng. 2019, 127, 25–30.

(21) Shamsuzzoha, M.; Skogestad, S. The setpoint overshoot method: A simple and fast closed-loop approach for PID tuning. J. Process Control 2010, 20, 1220–1234.

(22) Ziegler, J. G.; Nichols, N. B. Optimum settings for automatic controllers. Trans. ASME 1942, 433.

(23) Yadav, E. S.; Indiran, T.; Shankar, N. Optimal Actuation of Controller using Predictive PI for Nonlinear Level Process. Indian J. Sci. Technol. 2016, 9, 1–4.

(24) Yadav, E.; Indiran, T. Servo mechanism technique based anti-reset windup PI controller for pressure process station. Indian J. Sci. Technol. 2016, 9, 1–5.

(25) Yadav, E. S. Design implementation and validation of control schemes on the batch distillation column; 2020; https://shodhganga.inflibnet.ac.in/handle/10603/302186.