

Data-Driven System Identification and Closed-Loop Analysis of a Jacketed CSTR

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Abstract— This paper presents transfer function identification for the reaction of ethyl acetate hydrolysis with sodium hydroxide in a CSTR using simulation approach. The Ethyl acetate hydrolysis was simulated in a jacketed CSTR with 1.5 m in diameter and 4.5 m in height. Aspen HYSYS is used to simulate ethyl acetate hydrolysis with sodium hydroxide in a CSTR at dynamic simulation. MATLAB was used in determining the system transfer functions using system identification toolbox via black box technique for all system transfer functions, and then simulated the closed-loop response to conventional feedback temperature control for the CSTR and determining the system characteristics.

Keywords— Transfer function identification, System response, Dynamic simulation, Ethyl Acetate Hydrolysis, Aspen HYSYS, MATLAB.

I. INTRODUCTION

For efficient mixing, the height to diameter ratio (H/D) of the continuous stirred tank reactor (CSTR) is between 2 and 4. The reactor itself is a cylindrical container. frequently include baffles to improve mixing [1]. To guarantee that the reaction mixture is adequately combined, baffles are used. However, it prohibits any temperature or concentration gradients that are angular, radial, or axial. As a result, the temperature and concentration distribution within the CSTR is uniform, which leads to a consistent reaction rate across all locations within the CSTR [2]. The nonlinearity and lack of measurements of some industrial processes make challenge to control their systems to achieve their desired values [3-7]. Always, the nonlinear behavior of CSTR makes the system identification and control more difficult to establish. A control mechanism is needed to cancel the negative impact due to nonlinearities which may cause undesired effects in chemical plants. Generally, the increasing or decreasing in reaction temperature affects the reaction conversion, whereby, affecting the quality of the desired product. The controlling temperature guarantees the product quality in case of exothermic or endothermic reaction, or if there is any heat transferring from surrounding as disturbance [8]. It is necessary to build a strong and preventive control system to maintain the controlled variable at their desired values from any potential disturbances, such as fluctuation occurred from feed streams [9]. Whereby, it is important to identify the system transfer functions with knowledge of all state variables and parameters of the process.

All state variables and parameters of a nonlinear CSTR control system must be measured and estimated accurately.

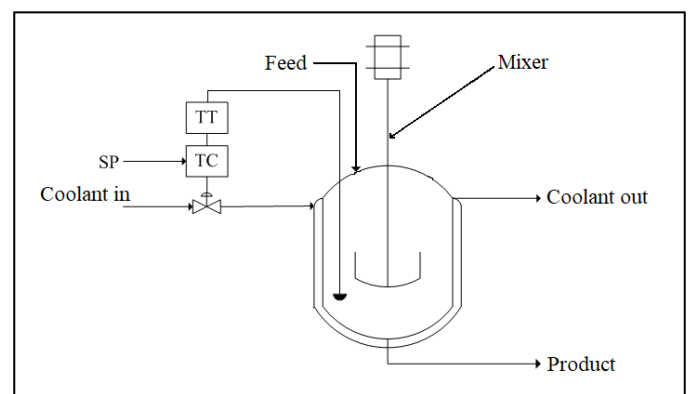
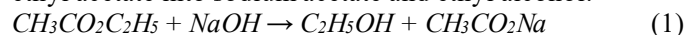


Figure 1: CSTR Temperature Control using Conventional Feedback System [10].

Antonelli and Astolfi [3], were mentioned that the chemical kinetic parameters uncertainty and unmeasured state variables may affect the stability of equilibrium points and yield model mismatch. Figure (1) shows a physical diagram for controlling temperature of CSTR with jacket using conventional feedback control system. The of feedback control system based on the error elimination between the desired set point and the controller variable by adjusting the manipulated variable automatically or manually the [9, 11].

The utilization of Aspen HYSYS to simulate ethyl acetate hydrolysis with sodium hydroxide in a CSTR at steady-state and dynamic simulation offers all state variables and parameters in order to model ethyl acetate hydrolysis.

[12, 13] demonstrates how sodium hydroxide breaks down ethyl acetate into sodium acetate and ethyl alcohol:



The reaction rate for the aforementioned ethyl acetate hydrolysis reaction is given as [13]:

$$-r = -r_{NaOH} = k C_{NaOH} C_{EtAC} \quad (2)$$

The reaction is a first order with respect to the sodium hydroxide and the ethyl acetate and it is the second order overall. This reaction is considered to be an equi-molar, non-catalytic, exothermic and an irreversible reaction [14]. Mukhtar et al. [15], studied the kinetics of ethyl acetate hydrolysis with sodium hydroxide at different temperatures and development of

mathematical model for holding time in batch reactor. They found the overall reaction order 1.3118 and cannot be expressed satisfactorily as a second order reaction, especially when equimolecular concentrations of both reactants are used. They found the activation energy equals to 4.409 kJ/mole, the heat of reaction (ΔH_{R298}) -36920 kJ/mol and Gibbs free energy (ΔG_{298}) -24100 kJ.

II. METHODOLOGY

Dynamic simulation study is essential to evaluate the flexibility of the cooling water volumetric flow rate with CSTR temperature and generate the data required for system identification. Based on the dynamic simulation analysis, a process control design would help in achieving the tight control of the temperature of the CSTR and maintain the stability of the process. The precision of dynamic simulation in AspenHYSYS imposes several extra requirements over steady-state mode, such as pressure-flow specifications and configurations of unit equipment's.

This paper was carried out using computational tools. Aspen HYSYS v 9.0 was used to provide a simulation model of ethyl acetate hydrolysis with sodium hydroxide. All physical property calculations for this reaction can be provided by this Aspen HYSYS v 9.0 process simulator, which define all information inside a single entity.

MATLAB R2018a software was used to identify the transfer functions for the CSTR temperature and the valve transfer functions of cooling water utility via steady-state and dynamic data obtained by HYSYS. In addition to this,

Therefore, MATLAB was used to simulate the System responses and determining the characteristics of the system

The research methodology employed for this project is summarized in figure (2) as shown below.

2.1 Steady-state Simulation

Sodium acetate was predicted by its molecular structure, while ethyl acetate, sodium hydroxide, ethanol, and water were chosen as the reacting components from the HYSYS database. weight, density, and typical boiling point. The property model Peng-Robinson-Stryjek-Vera (PRSV) model, which caters to real and very non-ideal (non-electrolytic) chemical systems, was chosen based on the considerations stated in the property package selection. The aforementioned reaction was defined in HYSYS by introducing a reaction set. The kinetic data were chosen from [13], where the frequency factor (k_0) and activation energy (E_a) were determined to be 2194760 and 41400 kJ/mol, respectively. First of all a steady state reaction was conducted. Both reactants entered the CSTR at 25 °C, 1 atm and 5 M. The feed flow rates for the both reactants were 5000 kg/hr. After clicking on the simulation environment tab, the material streams, energy stream and CSTR were installed in the simulation environment. The reactants and product streams conditions and the reactants streams compositions were entered. The simulated reaction system consisted of cylindrical CSTR configuration as shown in table (1). The energy stream was connected to the CSTR to maintain the reaction temperature at 30°C, which represented the cooling water jacket that cool down the reaction mixture in real CSTR.

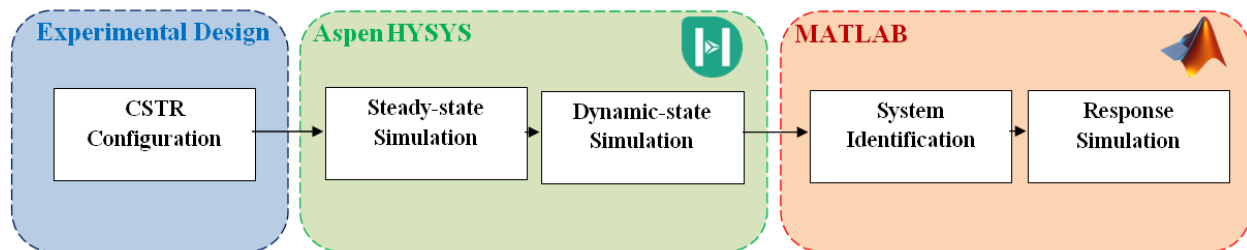


Figure 2: Flow Chart of Research Work.

TABLE 1: Summary of CSTR Design Calculations [16].

Dimensions	Value
Inside diameter (m)	1.50
Height (m)	4.50
Outside diameter (m)	1.51
Residence time (hr)	0.56
Volumetric feed rate (m ³ /hr)	10.00
Reactor volume (m ³)	7.50
Wall thickness (mm)	7.00
Jacket thickness (m)	0.10
Jacket type	Dimple jacket
Flat-end head thickness (mm)	2.15
Mass flow rate of cooling water (kg/hr)	30244.82
Agitator diameter (m)	0.50
Agitator blade width (m)	0.10
Agitator blade length (m)	0.13
Agitator height (m)	0.50
Speed of flat blade turbine (rpm)	150.00
Agitator power required (kW)	1.63
Material of construction	Stainless steel (304)

Besides that, the logical unit (SET-100) was added to adjust the mass flow rate of stream (S-2) by means of stream (S-1) using multiplier equals 1.00. The logical unit (SET-100) is used to avoid fluctuations that occurs when the simulation transfer from steady-state mode to dynamic mode. In order to define the specific heat capacity of cooling water utility, the inlet and exit cooling water temperatures were inserted to HYSYS by clicking on "Process Utility Manager" tab. The reaction set was coupled to the simulation environment, while the streams were connected to the CSTR.

In HYSYS, the concentration is in mole fraction basis rather than molarity. The conversion of molarity to mole fraction is given by formula [17]:

$$x = \frac{M(MW_2)}{M(MW_2 - MW_1) + 1,000d} \quad (3)$$

Where d = density of solution (g/l); M = molarity (mol/l); MW_1 = formula weight of solute (g/mol); MW_2 = formula weight of solvent (g/mol); x = mole fraction.

2.2 Dynamic Simulation

In this study, step response model is used for design of the proper controller. In order to get coefficients of step response model, it is necessary to obtain the open loop responses of controlled variables by giving unit step changes to the manipulated variables. The open loop responses of controlled variable, reactor temperature (T_R), for step input changes in the manipulated variable, cooling water volumetric flow rate. The simulation was converted into dynamic mode in order to get the controlled variable of CSTR temperature-time with step change response in cooling water mass flow rate. The integrator time was adjusted to real-time.

The valve opening was changed from 50% to 100% by means of step response change. Therefore, the changing in cooling water volumetric flow rate and CSTR temperature were recorded.

2.3 Transfer Function Identification

The transfer functions of the CSTR, valve and thermocouple sensor were identified using Transfer Function Toolbox that build-in MATLAB. The black box model was used for dynamic data obtained by Aspen HYSYS to determine the transfer functions.

III. RESULTS AND DISCUSSION

The ethyl acetate hydrolysis with sodium hydroxide was simulated in a CSTR using Aspen HYSYS according to the dimensions and the operating conditions those mentioned in subsections 2.2 and 2.3. The two feed streams, sodium hydroxide material stream (S-1) and ethyl acetate material stream (S-2) were defined as shown in figure (3). The cooling utility was defined as energy stream (Q-100). The exiting stream from the CSTR (S-4), is carried out the ethanol, sodium acetate, and the unreacted ethyl acetate, sodium hydroxide and water.

The dynamic data of the controlled variables and the steady-state data of the manipulated variables are shown in figures (4.3) and (2), respectively. Figure (4) shows the change in CSTR temperature with step change response of cooling water volumetric flow rate. Figure (5) shows the change in valve opening (%) with cooling water volumetric flow rate.

From figure (4), the volumetric flow rate of cooling water was changed from 0.16 to 0.34m³/min in dynamic mode to examine its response on the CSTR temperature. The change in reactor temperature was observed in changing from 39.3 °C, and gradually increased until the reactor temperature riched to 30.1 °C at 250 min as shown in figure (4).

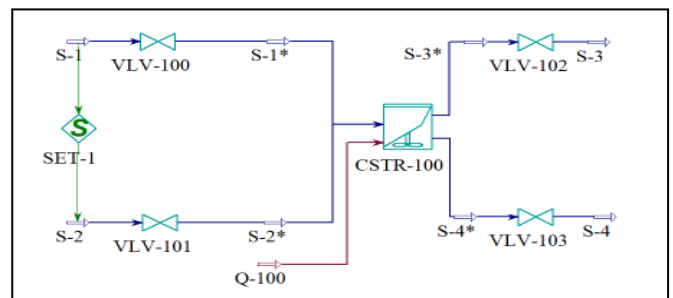


Figure 3: CSTR Flow Sheet in Aspen HYSYS Simulation Environment.

TABLE 2: Summary of Simulated Material Balance around the CSTR.

Materials	Molar flow rate (mol/hr)		
	S-1	S-2	S-3
Sodium hydroxide	24.73	-	0.99
Ethyl acetate	-	25.15	1.41
Sodium acetate	-	-	23.74
Ethanol	-	-	23.74
Water	222.57	154.50	377.07
Total	426.95	426.95	426.95

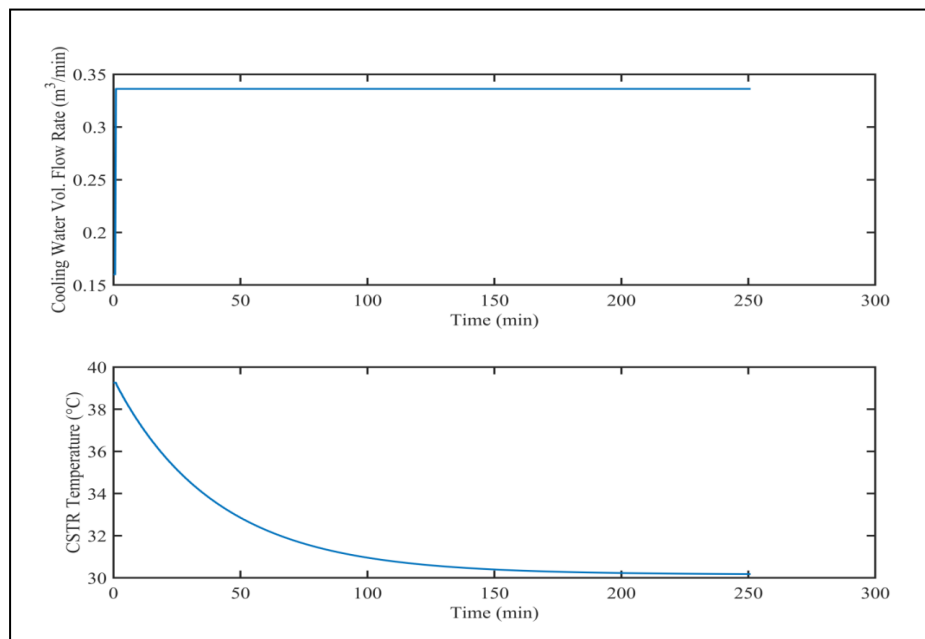


Figure 4: Response of CSTR Temperature with Cooling Water Volumetric Flow Rate Step Change.

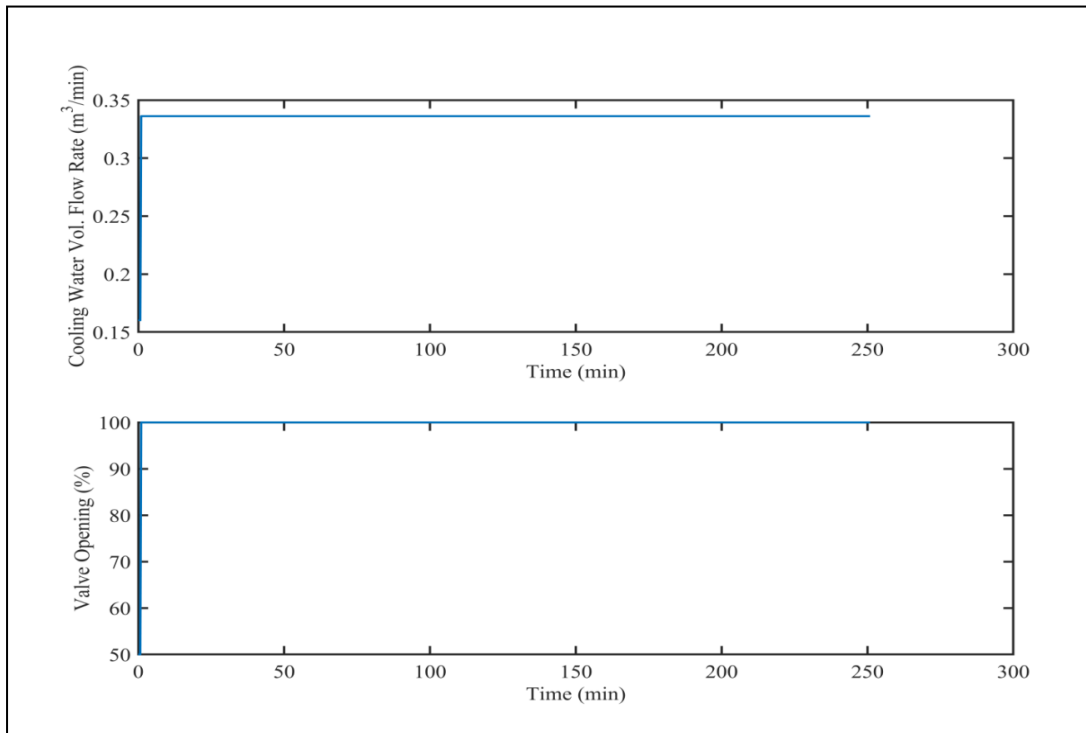


Figure 5: Response of Cooling Water Volumetric Flow Rate with Valve Opening step change.

In figure (5), the response of valve opening (%) on volumetric flow rate of cooling water utility was examined in dynamic-state mode. The step change from 50% to 100% of the valve opening, and the response of the cooling water volumetric flow rate was from 0.16 to 0.33 m³/min.

Using System Identification tool in MATLAB, the transfer function for the process, and the valve element are, respectively:

$$G_p = \frac{74.52}{(0.87s+1)(0.15s+1)} \quad (4)$$

$$G_v = \frac{0.028}{25.67s+1} \quad (5)$$

In HYSYS, it assumed the thermocouple is perfectly accurate in the CSTR temperature measurement [18]. Thus, the transfer function of the measuring element is:

$$G_m = 1 \quad (6)$$

Therefore, figure (6) showed the feedback control system for the CSTR with block diagrams of process, valve and measuring element.

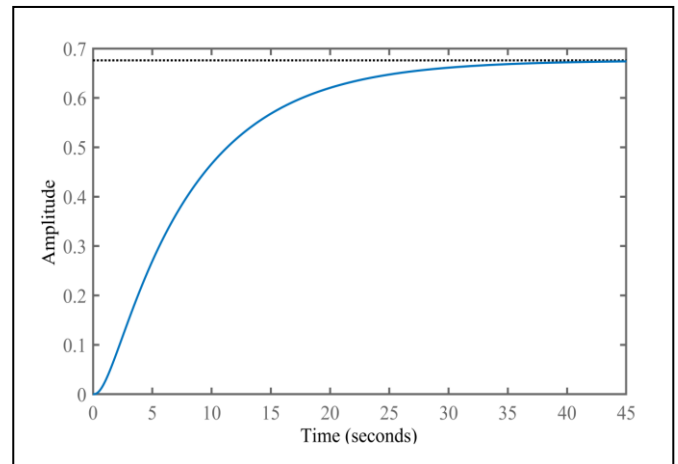


Figure 7: System Response for the Closed-loop.

TABLE 3: Characteristics of the Step Response of the Closed-loop.

Characteristic	Value
Overshoot (%)	0.00
Peak amplitude	≥ 0.674 (at > 45 sec)
Rise time (sec)	16.80
Settling time (sec)	30.70
Final value	0.68

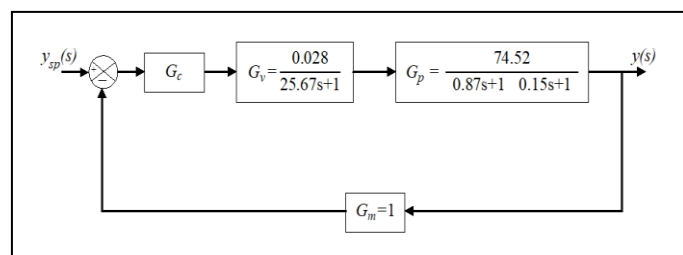


Figure 6: Block Diagram of Conventional Process Control.

The step response change simulation of the closed loop is shown in figure (7). The step response change simulation is shown the behavior of the closed-loop. The performance characteristics of the step response of closed-loop system are shown in table (3). Figure (7) shows the deviation from the desired value of 1.00. In order to achieving the desired value of P, step change, and increasing the rising time and settling time, P,

PI and PID controllers must be designed and simulated using step response change. Whereby, to identify the most suitable controller that gives the best performance with respect to minimum overshoot [19].

IV. CONCLUSION

Aspen HYSYS is implemented to simulate ethyl acetate hydrolysis with sodium hydroxide at steady-state and dynamic simulations using PRSV equation of state, according to CSTR configuration. The steady-state and dynamic data were used for identifying the CSTR, valve and thermocouple transfer functions using Transfer Function Identification toolbox. MATLAB was used for simulating the system response of the ethyl acetate hydrolysis.

REFERENCES

- [1] W. L. Luyben, *Chemical Reactor Design and Control*. USA: John Wiley & Sons, Inc., 2007.
- [2] H. S. Fogler, *Elements of Chemical Reaction Engineering*, 4th ed.: Pearson Higher Education, Inc., 2006.
- [3] R. Antonelli and A. Astolfi, "Continuous stirred tank reactors: easy to stabilise?," *Automatica*, vol. 39, pp. 1817-1827, 2003.
- [4] D. Zhao, Q. Zhu, and J. Dubbeldam, "Terminal sliding mode control for continuous stirred tank reactor," *Chemical engineering research and design*, vol. 94, pp. 266-274, 2015.
- [5] M. Kumar and R. Singh, "Comparison of non-linear, linearized 2nd order and reduced to FOPDT models of CSTR using different tuning methods," *Resource-Efficient Technologies*, vol. 2, pp. S71-S75, 2016.
- [6] B. Tandon, S. Narayan, and J. Kumar, "Nonlinear disturbance observer based robust control for continuous stirred tank reactor," *ICIC Express Letters*, vol. 12, pp. 1-7, 2018.
- [7] F. Wallam and A. Y. Memon, "A robust control scheme for nonlinear non-isothermal uncertain jacketed continuous stirred tank reactor," *Journal of Process Control*, vol. 51, pp. 55-67, 2017.
- [8] O. P. Verma, S. Kumar, and G. Manik, "Analysis of hybrid temperature control for nonlinear continuous stirred tank reactor," in *Proceedings of Fourth International Conference on Soft Computing for Problem Solving*, 2015, pp. 103-121.
- [9] M. Djaeni, Suherman, K. Jalsanti, and R. R. Mukti, "Control System Strategy Of The Saponification Process Between Ethyl Acetate And Sodium Hydroxide," *Reaktor*, vol. 5, pp. 54-58, 2001.
- [10] G. A. Gasemseed, *A Textbook of Chemical Engineering Process Control*. Khartoum, Sudan: G-Town Center, 2013.
- [11] J. Schork, *Control of Polymerization Reactors*: CRC Press, 2019.
- [12] A. Mukhtar, U. Shafiq, M. O. Qazi, H. A. Qadir, M. Qizilbash, and B. A. Awan, "Kinetics of Alkaline Hydrolysis of Ethyl Acetate by Conductometric Measurement Approach Over Temperature Ranges (298.15-343.15K)," *Austin Chem Eng*, vol. 4, p. 1046, 2017.
- [13] U. P. L. Wijayarathne and K. C. Wasalathilake, "Aspen Plus Simulation of Saponification of Ethyl Acetate in the Presence of Sodium Hydroxide in a Plug Flow Reactor," *International Journal of Chemical and Molecular Engineering*, vol. 8, pp. 1089-1096, 2014.
- [14] A. Afzal, I. A. Muhammad, Y. Muhammad, K. Hayat, and U. H. S. Mansoor, "A comparative study of alkaline hydrolysis of ethyl acetate using design of experiments," 2013.
- [15] A. Mukhtar, U. Shafiq, M. Qazi, H. Qadir, M. Qizilbash, and B. Awan, "Kinetics of alkaline hydrolysis of ethyl acetate by conductometric measurement approach over temperature ranges (298.15-343.15 K)," *Austin Chemical Engineering*, vol. 4, 2017.
- [16] M. H. H. Deifalla, "Simulation and Control of CSTR for Ethyl Acetate Hydrolysis Using Aspen HYSYS and MATLAB," M.Sc thesis, Chemical Engineering Department, University of Science and Technology, 2020.
- [17] J. H. Montgomery, *Groundwater Chemicals Desk Reference*, 4th ed.: CRC Press, 2007.
- [18] Aspen HYSYS, *Dynamic Modeling*. Cambridge, USA: Aspen Technology, Inc., 2004.
- [19] D. E. Seborg, T. F. Edgar, D. A. Mellichamp, and F. J. Doyle III, *Process Dynamics and Control*, 4th ed. USA: John Wiley & Sons, 2017.