

**EXPERIMENTAL VALIDATION OF MODEL-BASED CONTROL STRATEGIES FOR
MULTICOMPONENT AZEOTROPIC DISTILLATION****Lina Rueda, Thomas F. Edgar, R. Bruce Eldridge***Department of Chemical Engineering, University of Texas at Austin*

Abstract: This work presents the results from dynamic modeling and control of an azeotropic distillation system. The model was validated with experimental data from a packed distillation unit. The physically-based process dynamic model, developed in HYSYS, was linked online with the control software used in the process. Model parameters were modified online using a feedback configuration to eliminate the difference between the process and model outputs. The fundamental model was used in the implementation of different control strategies, including a multivariable control strategy using model predictive control (MPC) software Predict Pro, via an inferential control strategy to treat missing process measurements. *Copyright © 2006 IFAC*

Keywords: Dynamic modeling, distillation, model-based control, multivariable control.

1. INTRODUCTION

Distillation is clearly the largest energy-consuming separation process used in chemical industries to recover products, by-products and unreacted raw materials. Improving its process efficiency is an on-going goal of the chemical processing and refining industries, given recent increase in energy prices. The use of dynamic modeling software in chemical and refining applications has been intensified with the adoption of commercial process modeling software and increased computer processing capabilities. The modeling software is used in a broad range of applications like parameter estimation, process optimization, and control. Most modern control methods require some kind of process model to predict future process outputs but industrial applications typically do not link fundamental dynamic models in commercial software with the control software. Some model-based control and optimization techniques are based on steady state physical models that account for the physical drifting of the process itself (such as fouling of a heat exchanger, temperature fluctuation of the feed, etc.) or changes in market demands and economic conditions, information that can be used to modify product specifications and plant schedules.

Azeotropic distillation is a process widely used to separate non-ideal binary mixtures. This separation technique uses another component, known as an entrainer. Depending on the mixture, the entrainer forms an azeotrope with one of the components in the binary mixture or breaks an existing azeotrope in the binary mixture. There are three azeotropic distillation configurations: homogeneous azeotropic distillation, heterogeneous azeotropic distillation and extractive distillation. Azeotropic distillation presents multiple challenges in design and operation due to the presence of non-idealities, phase splitting, possible multiple steady states, and distillation boundaries. When designing these systems, it is important to keep in mind that distillation boundaries cannot be crossed. For these reasons, in order to isolate two pure components which lie in two different distillation regions, it is necessary to have two different feed compositions (one from each of the two regions) and two distillation columns (Doherty and Calderola 1985). Published experimental work on azeotropic distillation has often analyzed the behavior of the azeotropic distillation systems in laboratory scale sieve columns (Baur, *et al.*, 1999; Müller and Marquardt, 1997; Springer and Krishna, 2001; Wang, *et al.*, 1998); in addition, some experimental studies (Chien, *et al.*, 2000) have also implemented

different control strategies using temperature as the controlled variable.

This work presents the results from experimental validation of dynamic models of an azeotropic distillation system of methanol, normal pentane and cyclohexane. All the experiments were performed in the homogeneous region without liquid phase splitting. The model was validated with experimental data from a pilot-scale size packed distillation unit configured at finite reflux. The approach presented in this work links the process fundamental dynamic model (HYSYS) with the control software used in the process. The model is modified online using a feedback configuration to eliminate the difference between the process and model outputs. The model is used in the implementation of different control strategies to infer process variables that cannot be determined with field instrumentation. Two different variable pairings are studied and the results from individual control loop configurations are compared with a multivariable control strategy using model predictive control (MPC).

The dynamic model was developed using HYSYS from Aspen Technologies. The model was linked to Emerson Process Management's DeltaV digital automation system. The experiments were developed in the pilot plant of The Separation Research Program (SRP) at The University of Texas at Austin.

2. EXPERIMENTAL SYSTEM

The chemical system selected for the experiments performed in this research was a ternary mixture of methanol, pentane and cyclohexane. The ternary mixture diagram is presented in Figure 1 where the two azeotropes in the system are apparent. The two azeotropes divide the diagram into two distillation regions. Figure 1 also identifies the feasible product region for a particular feed point using the intersections between the distillation and material balances lines.

The highest pentane purity achievable in the distillate product was the azeotropic composition, which was a viable objective in both regions; however, the bottom composition objective changed from pure cyclohexane in the first region to pure methanol in the second.

The plant where the experiments were carried out is located at the Separation Research Program, at the University of Texas at Austin. The column used in the experiments is a 6 in stainless steel column with 30 ft of contacting height packed with 0.7 Nutter rings metal random packing.

The system is well-instrumented with state of the art sensors and actuators from Fisher-Rosemount. The experimental plant is operated with Emerson Process Management's DeltaV digital automation system. The simulation was implemented in HYSYS from Aspen Technologies at the application station in the DeltaV system and connected to the controllers through an interface with the digital control system. Additional details

on the equipment configuration can be found at the SRP website <http://uts.cc.utexas.edu/~utsrp/>.

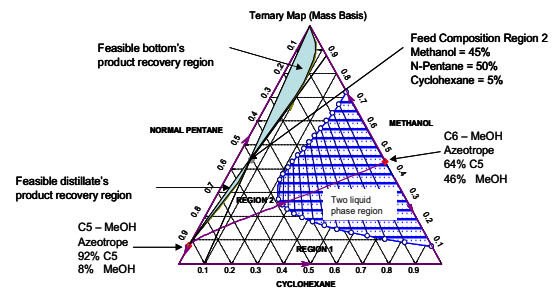


Fig. 1. Ternary map (mass basis) for cyclohexane, normal pentane and methanol. P = 6 psig. Property Package: Split from Aspen Tech.

An analytical procedure for the analysis of the samples collected from the system was developed and implemented in two HP 5890 gas chromatographs. The error in the measurement was calculated to be less than 3%.

3. AZEOTROPIC DISTILLATION DYNAMIC MODEL

The fundamental model was developed following a methodology that included five steps: (1) the system physical and thermodynamic behavior was identified and appropriate physical property relationships were determined; (2) different modeling approaches were studied and compared with process data to determine the most suitable method to model the system; (3) the model was developed and validated with process data; (4) the model parameters to be updated on-line were selected; and (5) the model updating method was implemented.

Steady state simulation and ternary and binary diagrams were used to study the system's thermodynamic behavior. After analyzing the ternary diagram and validating the simulated data with experimental data, it was concluded that the process did not display multiple steady states. The existence of multiple steady states could be determined based on the geometry of the distillation region boundaries and product paths in the ternary diagrams (Bekiaris *et al.*, 1993; Bekiaris and Morari, 1996).

Some studies have concluded that rate-based or non-equilibrium models are necessary to obtain a good description of the azeotropic system (Repke *et al.*, 2004; Springer and Krishna, 2001), while others have validated azeotropic distillation equilibrium models experimentally (Müller and Marquardt, 1997; Kumar *et al.*, 1984). These studies suggest that the equilibrium approach can perform very well in modeling of azeotropic distillation systems. In order to determine whether or not equilibrium models could be used to accurately predict the azeotropic system behavior, non-equilibrium and equilibrium steady state models were developed, and their model predictions were compared with a wide range of experimental data. Both models used the same

equipment configuration, operating conditions, and thermodynamic properties. Conditions from the two distillation regions were simulated, and their results were validated experimentally. It was concluded that the equilibrium model did accurately predict the azeotropic behavior and therefore a dynamic equilibrium model was developed.

Table 1. Column Configuration for Steady State Simulation.

Number of Theoretical Stages	24 (Without condenser and reboiler)
Feed Stage	18
Condenser Type	Total (Stage 1)
Reboiler Type	KETTLE (Stage 26)
Valid Phases	Vapor-Liquid-Liquid Packed (Nutter Ring)
Internal Type	Metal Random No. 0.7)
Stage Packing Height [in]	13.84615
Stage Vol [ft ³]	0.226557121
Diameter [in]	6
Void Fraction	0.977
Specific Surface Area [sqft/cuft]	68.8848
Robbins Factor	11.8872

The column configuration is summarized in Table 1. The activity coefficient model NRTL was used as the main property method for the liquid phase while the Redlich-Kwong equation-of-state was used for the calculations in the gas phase.

Analysis of the process dynamic responses indicated that in this particular system temperature was not a good choice as a controlled variable. The temperature response to feed disturbances and changes in the steam and reflux flow rates displayed a highly nonlinear behavior. In addition, in some regions the gain was very small and the changes in temperature due to changes in the manipulated variables were within the noise level.

4. ONLINE MODEL RECONCILIATION

The dynamic model was intended to be used as a tool to determine process parameters that could not be measured directly in the field, such as composition, a variable needed to implement the control strategies. For this reason, the model predictions had to be accurate and track the process behavior throughout the entire operating region. Traditionally, model reconciliation is performed using a steady-state model and the parameter estimates are obtained off-line using an optimization algorithm, such as the weighted least square (WLS) formulation, where the objective is to find estimates that minimize the squared error between the model predictions and the measurements, normalized by the measurement covariance. Usually before the parameters are estimated, the measurement data are first validated with some conservation equations, and then reconciled such that the model parameters and the adjusted data satisfy the process model equations (Seborg, *et al.*, 2004).

In this work we used a reconciliation module to calculate the model parameters that minimize the error between plant measurement and model variables. The algorithm used in the reconciliation module is based on the gradient approach for

model-reference adaptive control (Åström and Wittenmark, 1995). The objective is to modify the parameters in the model so that the error between the outputs of process and reference model is driven to zero. In the gradient approach the parameter is obtained as the output of an integrator. A quicker adaptation could also be achieved by adding a proportional adjustment to the integral action. The control law then takes the form of (1), which can be implemented in the plant using PI controller software where the constants γ_1 and γ_2 represent the proportional and integral gains respectively (Åström and Wittenmark, 1995).

$$u(t) = \gamma_1 e(t) + \gamma_2 \int_0^t e(\tau) d\tau \quad (1)$$

Initially, the model parameters selected to be updated online were overall column heat transfer coefficient and HYSYS dynamic efficiency (Abouelhassan and Simard, 2003). Given that the packing HETP value was obtained from experimental data, the efficiency value should not change considerably from the value of one. However, it was expected to have some variations given different flooding conditions mainly due to the system's non-ideal behavior. The model efficiency value was modified to match the process distillate C5 composition. Because the error in the measurement was about 3%, the parameter was modified if the model output was off by more than 3% from the process output. After data from the experiments was analyzed, it was concluded that the efficiency value was fairly constant at a value of 0.7 in the distillation region rich in cyclohexane and pentane and 0.5 in the distillation region rich in methanol and normal pentane. Although the reconciliation module was used to determine these values, the efficiency parameter was not longer modified on-line to reconcile the model on-line.

The column's external surface heat transfer coefficient directly influences the heat loss experienced by the column. The model developed in this work used a simple heat loss equation, where the heat loss is calculated from the parameters specified by the user: overall heat transfer coefficient U and ambient temperature T_{amb} . The heat transfer area A and the fluid temperature T_f are calculated for each stage by the model. The heat loss is calculated using (2).

$$Q = UA(T_f - T_{amb}) \quad (2)$$

During the model validation phase, U was modified until the mass balance in the model matched the experimental mass balance, that is distillate and bottoms flow rates were the same in the model and the experiment when all the other conditions in the model were set to match the conditions in the experiment. T_{amb} was introduced given the conditions of the experiment, but was not continuously upgraded. The heat transfer coefficient was updated online using the reconciliation module during the control experiments described in the next section. Its value

increased up to 5% as the liquid flow in column decreased and vice versa. The heat transfer coefficient is dependent upon the physical properties of the fluid and the physical conditions of the experiment, since both fluid composition and process conditions changed with the operation region the heat transfer coefficient also changed. In addition, the heat transfer coefficient was reflecting the variations in the ambient temperature given that this value was not measured continuously nor automatically upgraded during the experiments. Although this variation was found to be small, it shifted the model from the process outputs.

5. CONTROL STRATEGIES

Stabilizing the basic operation of the column was achieved by inventory (level), flow and pressure controls. The control loops in this level were configured with independent PID controllers (See Table 2).

Table 2. Basic Configuration

Manipulated Variable	Controlled Variable
Feed Flow Valve Position	Feed Flow Rate
Preheater Steam Flow Valve Position	Feed Temperature
Reflux Flow Valve Position	Reflux Flow Rate
Distillate Flow Valve Position	Distillate Flow Rate
Bottom Flow Valve Position	Bottom Flow Rate
Reboiler Steam Flow Valve Position	Steam /Duty Flow Rate
Nitrogen Flow Splitter Valve Position	Column Pressure

To control the product composition in the column, two different configurations were considered based on the relative gain array analysis (RGA). RGA was used to get an initial understanding on how to pair variables in the inventory and separation control. The gain matrix was calculated using the step responses from the dynamic model developed in HYSYS. Different step changes were performed in the manipulated variables, using different magnitudes and directions, and then the results were averaged. The analysis indicated that two configurations were viable (see Table 3).

Table 3. Composition Manipulated and Controlled Variable Configurations.

	Manipulated Variable	Controlled Variables	Λ (RGA)	
1	Reflux Flow Rate (R)	DC - R	0.972	0.028
	Steam Flow Rate (Q)	BC - Q	0.028	0.972
2	Distillate Flow Rate (D)	DC - Q	-0.004	1.004
	Steam Flow Rate (Q)	BC - D	1.004	-0.004

DC = Distillate Composition; BC = Bottom Composition

The results from the RGA analysis were consistent with the traditional control configuration used in ordinary distillation (pairing 1) and the results from studies in azeotropic distillation where the opposite pairing (pairing 2) gave less loop interaction than the traditional variable pairing used in distillation (Chien, *et al.*, 2000; Tonelli, *et al.*, 1997).

As mentioned previously, the process had two feasible distillation regions. The data presented in this paper includes experimental data only from region one (feed composition with high concentration of cyclohexane and normal pentane). The control objective was to maintain the pentane/methanol azeotrope in the distillate and maximum recovery of cyclohexane in the bottom stream. For this reason the key components selected for control were normal pentane for the distillate stream and cyclohexane for the bottom stream. The manipulated variables were selected between the same options as for inventory control: distillate, reflux, steam, and bottom flow rate.

The level in the reflux drum was paired with the distillate flow rate in the first configuration (pairing 1) and with the reflux flow rate in the second configuration (pairing 2). The column level was paired with the bottom flow in the two control configurations.

The dynamic model was connected online to the DCS and provided estimates for variables where instrumentation was not available. Since the plant did not have an online measurement of composition, this configuration provided the controlled variable estimates. During experimentation, samples of distillate and bottom products were collected after mass balance was achieved in the process and compared with the values provided by the simulation. The difference between measured and estimated values was within $\pm 3\%$. Samples of the feed were collected every half hour and the values introduced in the model.

PID controllers were configured in the experimental plant to control the composition in the distillate and bottom streams using the pairings described in Table 3. The tuning of the PID controller was performed using the advanced control module DeltaV Tune, which implements a relay oscillation test based on the Aström-Hägglund algorithm for calculating the tuning parameters of a process control loop (Seborg, *et al.*, 2004). The results are given in Table 4.

Table 4. Composition Controller Tuning.

	Pairing 1		Pairing 2	
	DC - R	BC - Q	DC - Q	BC - D
Ultimate κ	10.90	10.55	6.42	4.98
Ultimate T	207.00	699.50	663.50	277.50
Process θ	28.45	85.91	99.46	42.98
Process κ	0.72	0.82	1.14	1.43
Process τ	257.42	957.85	766.61	311.92
Suggested Tuning Parameters:				
PID	P: 2.31	P: 1.66	P: 0.86	P: 1.15
	I: 191.1	I: 654.16	I: 369.39	I: 227.13
	D: 30.58	D: 104.67	D: 59.1	D: 36.34
θ Dominant	P: 2.72	P: 2.64	P: 0.79	P: 1.25
	I: 52.78	I: 178.37	I: 165.24	I: 70.76
Implemented Tuning Parameters:				
θ Dominant	P: 2	P: 2	P: 0.5	P: 1
	I: 191	I: 654	I: 369	I: 227
	D: 30	D: 104	D: 59	D: 36

κ = Gain. θ = Dead Time. τ = Time constant. T = Period.

Although the steam loop exhibited a considerable dead time that could limit the effectiveness of the controllers, based on experimentation it was

determined that the best PID tuning parameters were close to values suggested in the literature. The response with the dead time dominant configuration was more aggressive and exhibited oscillatory behavior.

5.1 PID Controller Performance

Figure 2 illustrates the PID controller performance after a series of step changes in the distillate and bottoms composition set points.

Both controllers drove the controlled variables to the desired set point. Pairing 2 gave fast responses but presented poor rejection to interaction. Figure 3 illustrates the closed-loop responses to disturbances in the feed temperature.

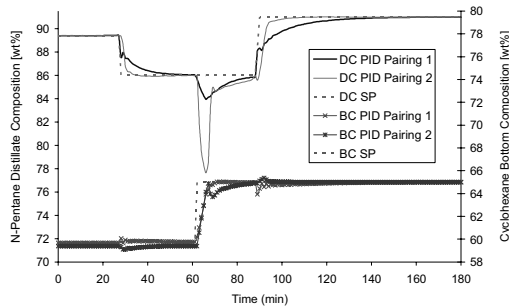


Fig. 2. Closed-loop composition control using PID controllers.

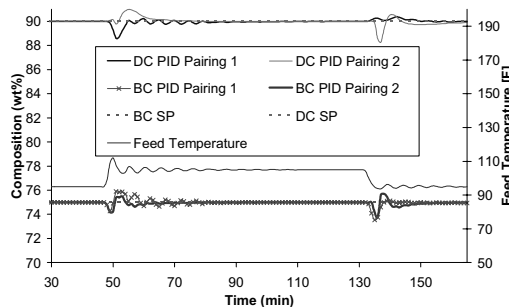


Fig. 3. PID controller closed-loop response to disturbances in the feed temperature.

5.2 Model-based control

Linear MPC was implemented using the commercial advanced control module Predict Pro from DeltaV. The process model used by the controller was identified online using the process model identification tool included in the module. Although with DeltaV PredictPro it is possible to run an automated test on the process, a manual test was performed for each input variable to generate the data for model identification. DeltaV PredictPro uses step response modeling for the generation of the MPCPro controller.

The step responses are generated using two types of models: Finite Impulse Response (FIR) and Auto-Regressive (ARX). The FIR model is used to identify the process delay used in the ARX model. The identified step responses are presented in Table 5. The MPC variables were selected based on best result from the PID study.

The gain (κ) is dimensionless because it is normalized by the transmitter range. The controller in the MPC algorithm is designed as an online-horizon optimization problem that is solved subject to the given constraints.

Table 5. MPC Step response models.

	Distillate C5 Composition	Bottom C6 Composition
Reflux Flow rate	$\kappa = 3.8$	$\kappa = -1.4$
	$\theta = 16$ s	$\theta = 8$ s
Steam Flow rate	$\tau = 689.23$ s	$\tau = 172.31$ s
	$\kappa = -3.2$	$\kappa = 3.5$
Feed Temperature	$\theta = 48$ s	$\theta = 40$ s
	$\tau = 1828.95$ s	$\tau = 1899.86$ s
Feed Flow rate	$\kappa = -0.2$	$\kappa = 0.2$
	$\theta = 16$ s	$\theta = 88$ s
	$\tau = 344.62$ s	$\tau = 190.67$ s
	$\kappa = 0.4$	$\kappa = -0.2$
	$\theta = 24$ s	$\theta = 16$ s
	$\tau = 689.23$ s	$\tau = 221.54$ s

κ = Gain. θ = Dead Time. τ = First order time constant. Time to reach steady state= 960 s.

For MPC based on linear process models, both linear and quadratic objective functions can be used (Qin and Badgwell 2003). Equation (3) represents the control law that minimizes a quadratic objective function.

$$\Delta U(k) = (S^T Q S + R)^{-1} S^T Q \hat{E}^0(k+1) \quad (3)$$

The vector $\hat{E}^0(k+1)$ corresponds to the predicted deviations from the reference trajectory when no further control action is taken; this vector is known as the predicted unforced error vector. The matrices Q and R are weighting matrices used to weight the most important components of the predicted error and control move, vectors respectively (Seborg, *et al.*, 2004). In DeltaV Predict Pro the elements of Q are known as penalty on error while the entries of R are the “penalty on move”. The MPC controller is tuned by modifying the values of the matrices Q and R. R offers convenient tuning parameters because increasing the values of its elements reduces the magnitude of the input moves, providing a more conservative controller.

Figure 4 illustrates the linear MPC performance in the experiment after a series of step changes in the distillate and bottoms composition set points. Both output errors were assigned a penalty of one. The penalty on move was set to 25 for the steam flow rate and 20 for the reflux flow rate. In the experiment the optimizer was also configured to maximize the concentration of C5 in the distillate. The SP was allowed to change 0.5% for both controlled variables.

Given that the system is nonlinear, the tuning parameters in the multivariable controller were set up to provide robustness and eliminate oscillation in the response. The main difficulty occurred due to changing gains in the process. The gains related to the distillate composition were smaller when the azeotropic composition was reached in the distillate composition than in other regions with lower pentane recovery in the overhead product.

Figure 6 illustrates MPC responses inside and outside the azeotropic region with different tuning parameters.

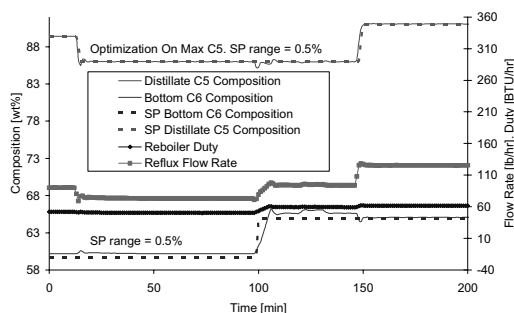


Fig. 4. Experimental composition control using linear MPC. Feed Flow Rate configured as manipulated variable.

Controller tuning 1 has a higher penalty on move (PM) for both manipulated variables than controller tuning 2. The parameters used in controller tuning 2 were the values suggested by DeltaV Predict Pro. These values are calculated based on the assumption that the system is linear. A higher penalty on move improved system stability in the region with higher gains. The penalty on error was set to 1 for both controlled variables. From Figure 5 it is observed that controller tuning 2 produces an unstable closed loop response.

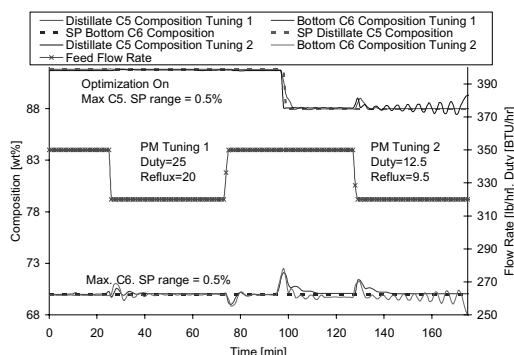


Fig. 5. Experimental MPC behavior using different tuning parameters.

6. CONCLUSIONS

Analysis of the process steady and dynamic models indicated that equilibrium models accurately predict the distillation column behavior. In multicomponent azeotropic distillation, temperature measurements do not offer accurate indications of composition, hence commercial dynamic simulation software were used to obtain an inferential control solution. Linear MPC gives excellent performance when the composition is used directly as a controlled variable and the appropriate tuning is used.

REFERENCES

Abouelhassan M., Simard A. (2003). The Scoop on Tray Efficiency. *Distillation Series, AspenTech Documentation*.

Åström K.J and Wittenmark B. *Adaptive Control* (1995). Addison-Wesley Publishing Co. Boston, MA.

Baur, R., Taylor, R., Krishna, R. and Copati, J. A. (1999). Influence of mass transfer in distillation of mixtures with a distillation boundary, *Trans IchemE, Part A, Chem Eng Res Des.* 77, 561–565.

Bekiaris, N., G.A. Meski, G.A. Raudu and M.Morari (1993). Multiple Steady States in Homogeneous Azeotropic Distillation. *Ind. Eng. Chem. Res.* 32(9), 2023–2038.

Bekiaris, N. and M. Morari (1996). Multiple Steady States in Distillation: 1=1 Predictions, Extensions, and Implications for Design, Synthesis, and Simulation. *Ind. End. Chem. Res.* 35, 4264–4280.

Chien, I-L., Wang, C. J., Wong, D. S. H., Lee, C.-H., Cheng, S.-H., Shih, R. F., Liu, W. T. and Tsai, C. S. (2000). Experimental investigation of conventional control strategies for a heterogeneous azeotropic distillation column. *Journal of Process Control*, 10(4), 333-340.

Doherty, M. F. and Calderola G. A. (1985). Sequencing of Columns for Azeotropic and Extractive Distillation. *Ind. Eng. Chem. Fundam.* 24, 474-485.

Kumar, S., Wright, J. D. and Taylor, P. A. (1984). Modeling and dynamics of an extractive distillation column. *Canadian Journal of Chemical Engineering.* 62(6), 780-9.

Müller, D. and Marquardt, W. (1997). Experimental Verification of Multiple Steady States in Heterogeneous Azeotropic Distillation. *Ind. Eng. Chem. Res.* 36, 5410-5418

Qin, S.J. and Badgwell, T.A. (2003) A Survey of Industrial Model Predictive Control Technology. *Control Eng. Practice.* 11, 733.

Repke, J-U, Villain, O. and Wozny, G. (2004). A nonequilibrium model for three-phase distillation in a packed column: modelling and experiments. *Computers & Chemical Engineering.* 28(5), 775-780.

Seborg, D. E., T. F. Edgar, and D. A. Mellichamp (2004). *Process Dynamics and Control*, Wiley, New York.

Springer, P. A. M. and Krishna, R. (2001). Crossing of boundaries in ternary azeotropic distillation: Influence of interphase mass transfer, *Int Commun Heat Mass.* 28, 347–356.

Tonelli, S. M., Brignole, N. B. and Brignole, E. A. (1997). Modeling and control of an industrial azeotropic train. *Computers & Chemical Engineering* 21(Suppl., Joint 6th International Symposium on Process Systems Engineering and 30th European Symposium on Computer Aided Process Engineering, 1997), S559-S564.

Wang, C. J., Wong, D. S. H., Chien, I-L., Shih, R. F., Liu, W. T. and Tsai, C. S. (1998) Critical Reflux, Parametric Sensitivity, and Hysteresis in Azeotropic Distillation of Isopropyl Alcohol + Water + Cyclohexane. *Ind. Eng. Chem. Res.* 37, 2835-2843.