

**A New Framework for Dynamic Parameter Estimation and Optimization of
Batch Distillation Columns**

Seyed Mostafa Safdarnejad

Jonathan R. Gallacher

J. D. Hedengren

Chemical Engineering Department

Brigham Young University

Approximately 40,000 distillation columns are used in different industries in the US that account for 6% of the yearly US energy consumption [1]. Continuous distillation columns are generally used in large scale production facilities; optimization of these large-scale units has been considered widely in the literature. Smaller-use items are often processed in batch distillation columns. Optimization of batch distillation columns has gained less attention in the literature, mainly because of the transient nature of both the columns themselves and the market for smaller-use items [2]. Recently, more research studies have considered optimization of the batch distillation columns [3]–[6]. The first step in optimization of batch distillation columns is to develop a model. Several research studies have considered model development for batch distillation columns. The models studied in the literature are generally categorized as rigorous [7]–[9] or shortcut methods [10]–[12]. Rigorous models generally include detailed descriptions of mass transfer, energy transfer and thermodynamics, and are therefore able to account for system non-idealities. While these methods are more accurate, they have high computational costs. In shortcut models, several simplifying assumptions have been considered that decrease the computational time. However, lack of experimental results makes it difficult to determine the accuracy of these models, restricting their use to ideal systems. In this study, a third approach is used to develop a model that incorporates some of the non-idealities of the process not seen in the typical shortcut methods without increasing the computational cost. In this approach, a model is developed by fitting parameters to simplified forms of shortcut models. The use of fitting parameters incorporates system non-idealities into the model but requires experimental data to determine parameter values. Dynamic parameter estimation is used to reduce the experimental load. The accuracy of the parameters is then determined by implementing nonlinear statistics calculations as well as a sensitivity analysis. These calculations help analyze the number of parameters that can

be estimated from the experimental data and return an acceptable range for those parameters. The framework used in this study is developed in a general approach that is applicable to other batch processes. The framework includes the use of ℓ_1 -norm dynamic parameter estimation, nonlinear statistics, parameter sensitivity analysis, and dynamic optimization. These techniques have not been applied together to batch distillation columns before in a holistic approach to dynamic optimization. The results obtained from dynamic optimization are also validated in this study.

The batch column used to collect data is a 38 tray, 2 inch, vacuum-jacketed and silvered Oldershaw column. A constant heat input is supplied from a reboiler heater. The reboiler is initially charged with 1.5 L of a 50/50 wt% mixture of methanol and ethanol for each run, with the goal being a product of 99 wt% methanol. The manipulated variable is the reflux ratio. A doublet test is implemented to collect data for model validation. Initially four parameters are assumed for fitting data to the model. These parameters are heater efficiency, vaporization efficiency, condenser molar holdup, and tray molar holdup. The maximum error observed between the model prediction and experimental data for the instantaneous distillate is 10%. For all but four measurements the model prediction is within 2%. The error between the prediction and measurement for the cumulative number of methanol is negligible.

Although the fit between the model and experimental data is excellent, a glance at the confidence interval reveals a wide region for all parameters except the heater efficiency. This might be because of the over-parametrization of the problem. A sensitivity analysis is therefore used to determine the number of the parameters that best represent the model. The singular value decomposition of the sensitivity matrix is implemented and it is found that there is one principle parameter (heater efficiency) that can be used to match the production data (number of moles of methanol). The singular value analysis also reveals that only one other parameter is required to

estimate the composition of methanol. In this study, vaporization efficiency is selected as the second parameter and the other two parameters (condenser and tray holdups) are assumed fixed for further analysis. Implementing the confidence interval calculations for the model with two parameters shows a narrow confidence interval that confirms the results obtained from the sensitivity analysis. The model prediction with two parameters also match the experimental results.

The validated model is then used to maximize the amount of methanol production for a 90 minute batch time. The non-optimized base case production over a 90 minute run is 9.5 moles of 99.2 mol% methanol at a constant reflux ratio of 4. To optimize methanol production, reflux ratio is varied every 5 minutes by the optimizer. Optimization results show that the reflux ratio starts low, then increases in a nominally linear pattern. This is done to take advantage of the high methanol concentration at the top of the column after the startup period. The model prediction for the optimization case is 9.75 moles of 99.0 mol% methanol. Implementing the optimized reflux ratio in the batch column resulted in 10.8 moles of 99.8 mol% methanol obtained in the experiment. The difference between model prediction and experiment is 10% and 0.8% for overall production and product composition, respectively. The agreement between model and experiment is excellent and reflects the work done to validate the model. Methanol production using the optimized reflux ratio represents a 14% increase in column production over the base case with a reflux ratio of 4. The increase in the productivity of the column highlights the potential benefits in applying the proposed framework in larger batch distillation columns as well as other batch processes.

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