CHEMICAL ENGINEERING TRANSACTIONS
VOL. 29, 2012
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at: www.aidic.it/cet
Guest Editors: Petar Sabev Varbanov, Hon Loong Lam, Jiří Jaromír Klemeš

ISBN 978-88-95608-20-4; **ISSN** 1974-9791
DOI: 10.3303/CET1229055

Predicting Minimum Energy Conditions for a Distillation Column by Design of Experiments and Process Simulation

Adela Drljo*, Bettina Liebmann, Walter Wukovits, Anton Friedl

Vienna University of Technology, Institute of Chemical Engineering, Getreidemarkt 9/166, 1060 Vienna, Austria
adrljo@mail.tuwien.ac.at

Distillation is the most commonly used and the most versatile separation method for liquid components in boiling mixtures. Unfortunately, this unit operation is often one of the biggest energy consumers in industrial processes. The energy consumption of the distillation column is dependent on several operation variables; optimization of these variables means to minimize the energy demand while maintaining good product quality. In the classical optimization approach, only one variable is varied at a time, and its effect on the system is recorded. This so-called “univariate” approach often requires a considerable experimental or computational effort, and it neglects relationships between the variables. In the multivariate optimization approach, the variables are varied in a more efficient way, and their possible interaction is taken into account. In the present work, a multivariate approach is used to define the optimal operation variables for minimum energy consumption of a distillation column. In the studied case, the distillation column is the solvent recovery in an organosolv process. The system is set up in the process simulation software Aspen Plus. In the given set-up, three independent column variables are identified for optimization: number of column stages, feed stage location, and solvent concentration at the column top. The multivariate optimization is performed in software Design Expert, and combined with process simulation results. The result of the multivariate approach is an empirical regression model for calculating the energy demand of the column and the optimum operation conditions.

1. Introduction

In the last decades, the efficient use of energy has become a highly relevant issue in industrial sector, since energy prices as well as environmental awareness are increasing. Therefore, industry is highly interested in procedures to analyze and optimize the energy consumption of processes. In particular, distillation is known as one of the most energy intensive units, and therefore needs to be carefully designed and operated in order to minimize energy utilization per unit of product (Fazlali et al., 2009).
According to the distillation theory, energy consumption is influenced by several factors, such as: appropriate feed stage location, number of column stages, reflux ratio, distillate flow rate, and product stream composition. In process simulation software, it is possible to vary only one variable at the time, and investigate its influence on the energy consumption (Wang and Li, 2010). This approach is not ideal for several reasons; three of them are: (1) it is necessary to perform numerous simulations to find the optimal set-up configuration, which results in low energy demand, (2) it usually misses the interaction effects of column variables, and (3) it does not result in a mathematical model that clearly describes the relationship between column variables and the resulting energy consumption of the column. Hence, introducing a multivariate approach in distillation optimization can overcome all the

Please cite this article as: Drljo A., Liebmann B., Wukovits W. and Friedl A., (2012), Predicting minimum energy conditions for a distillation column by design of experiments and process simulation, Chemical Engineering Transactions, 29, 325-330

mentioned shortcomings, and improve the procedure (Wang et al. 2012). In this work, a combination of process simulation and design of experiments, applying software Aspen Plus and Design Expert, is used in order to efficiently optimize a distillation column. Process simulation software Aspen Plus is used to simulate the distillation process and to calculate the column's energy consumption at the different conditions. These conditions are, in fact, the different values of three column variables, which are efficiently chosen by the software Design Expert. In this work, the so-called "experiments" are the simulation runs in Aspen Plus at different distillation conditions; the "response" of the system is the energy demand of the distillation column. At the end, all data provided from the simulation are analyzed by the "response surface methodology", and an empirical regression model for predicting the energy demand of the column is built.

2. Methods

2.1 Aspen Plus simulation of the distillation column

The investigated distillation column is part of an organosolv biorefinery process. The feed enters the distillation column with a total mass flow of approximately 19000 kg/h, and known composition of approximately 28 % w/w of ethanol, of which 99 % have to be recovered and recycled. In addition to ethanol, the feed stream consists of water and dissolved organic and salt compounds.

The system is set up in Aspen Plus (V7.1, Aspen Technology, Inc., Burlington, USA, 2008) simulation software, using the ready-made model "RadFrac" for the distillation column (Figure 1). The thermodynamic properties of the ethanol-water mixture are calculated based on the ELECNRTL property method. Since the maximum temperature allowed in the process is 70 °C, the distillation is operated under vacuum conditions. The reflux ratio was adjusted by the design specification to achieve 99 % recovery of ethanol from the feed. For complete definition of the distillation column, the following three variables remain: number of stages (N), feed stage location (FSL), and distillate rate. Note that the distillate rate is directly proportional to the ethanol concentration at the column top (CT).

Reasonable ranges for these variables are determined in preliminary calculations, and define the boundary conditions as follow: The number of feed stages (N) in the column can vary from 10 to 26. The feed stage location (FSL) values can range from 20 % to 80 %. The stage where the feed enters the column is given in percent of the total number of stages, and is counted from the top of the column. For example, 20 % FSL in a column with N=10 stages means that the feed enters at the stage number 2, counted from the top. The top concentration of ethanol can vary from 75 % to 90 % w/w of ethanol. Once all three variables are provided, the energy consumption of the column can be calculated in kJ/kg of ethanol.

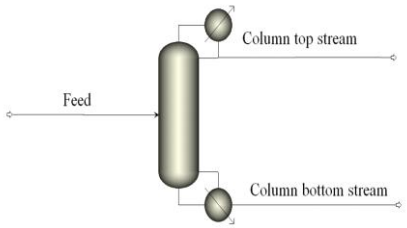

Figure 1: Aspen Plus process scheme of the distillation column for the ethanol recovery in organosolv process

2.2 Design of Experiments and Response Surface Methodology

Design of experiments (DoE) is a systematic approach to analyze systems/processes that are influenced by many variables (Jacquez, 1998). DoE is efficient, because it manages an adequate analysis with the least amount of experiments. Different types of experimental designs are available in software Design Expert (Version 6.0.11, Stat- Ease, Inc., Minneapolis, 2005). The non-linear relation between the three distillation column variables (N, FSL, CT) and the response of the system (energy demand), motivates the choice of a D-optimal design. The variables are systematically varied on 5 different levels within the boundary conditions calculated beforehand (see section 2.1). As a result, the D-optimal design suggests performing 56 different "experiments" with given values of the variables. For the next step, one has to revert to Aspen Plus software to actually perform the 56 computational experiments, and calculate the 56 (different) values for the specific energy demand (in kJ/kg). The relationship between the variables and the response is then explored by the Response Surface Methodology (RSM) in

Design Expert. Based on a limited number of designed experiments, RSM approximates the "global" response for the investigated range of variables by a mathematical model (Baş and Boyaci, 2005). The advantage of this model is not only that it describes the system, but it also facilitates the minimization of the response by mathematical optimization.

3. Results and discussion

For the ethanol recovery case study, 56 simulation scenarios are suggested by D-optimal design in Design Expert. These 56 scenarios have different values for the three independent variables, that are (1) the number of column stages (N), (2) the location of the feed stage (FSL), and (3) the ethanol concentration at the column top (CT). Each of the variables is changed in 5 different levels (values).

3.1 Model for the energy demand of distillation

The Aspen Plus simulations of the defined 56 distillation scenarios result in energy values between 1700 and 71,900 kJ/kg. Since this is a very wide range, the energy data is converted by the base 10 logarithm to a more manageable range for the following regression analysis in Design Expert. The final regression model to predict the energy demand on logarithmic scale, $\log_{10}(E)$, is a cubic model including not only the three original variables, but also their squares as well as double and triple interactions (Eq.1).

$$\log_{10}(E) = 20.5+0.4\cdot N - 0.6\cdot CT - 0.1\cdot FSL + 9.9\cdot10^{-4}\cdot N^2 + 5.0\cdot10^{-3}\cdot CT^2 - 1.0\cdot10^{-3}\cdot FSL^2 - 6.0\cdot10^{-3}\cdot N\cdot CT - 4.7\cdot10^{-3}\cdot N\cdot FSL + 6.0\cdot10^{-3}\cdot CT\cdot FSL - 1.5\cdot10^{-5}\cdot N\cdot FSL^2 - 6.2\cdot10^{-5}\cdot CT^2\cdot FSL + 1.8\cdot10^{-5}\cdot CT\cdot FSL^2 + 8.1\cdot10^{-5}\cdot N\cdot CT\cdot FSL \quad (1)$$

Due to the nature of the distillation system, this high complexity of the model is necessary. Without the interaction terms, e.g., double interaction CT*FSL or triple interaction CT*FSL², the model would not be able to properly describe the energy demand. Thus, a univariate approach would be highly inadequate, as such interactions are never considered.

To quantify the model performance, a so-called test set validation is used: 25 additional sets of variable values are chosen, and the corresponding energy demand is calculated by the model as well as in Aspen Plus. The squared correlation coefficient between the predicted model values, E_{MOD}, and the simulated Aspen Plus values, E_{SIM}, is $R^2 = 0.959$, which indicates a good model performance. Figure 2 compares the energy demand calculated by the regression model (E_{MOD}) and the one calculated by process simulation (E_{SIM}). Most data points lie close to the dotted 45° line that indicates perfect agreement.

The difference (E_{SIM}-E_{MOD}) is referred to as errors or residuals. Typically, single errors are used to calculate the errors mean value, μ= 112 kJ/kg and standard deviation, σ=1107 kJ/kg. The confidence interval for 95 % of the model errors is μ+-2σ (Figure 3) and it seems unacceptably high. A closer look on the errors reveals that they are occurring at very high energy demand, primarily above 10000 kJ/kg. For example, for the highest simulated energy demand E_{SIM}=71899 kJ/kg, the model predicts E_{MOD}=70607 kJ/kg. The prediction error for this case is (E_{SIM}-E_{MOD}) = 1292 kJ/kg, an error within the 95 % confidence interval. The relative error compared to the simulated energy demand, E_{SIM}, is less than 2 %.

Since the model should help in saving energy, the region below 10000 kJ/kg energy demand is of paramount interest in this work. In this region, the model predicts very well, and has generally small errors (Figure 3). Practically, the most relevant region of energy demand is estimated to be below 2000 kJ/kg. This energy threshold is estimated on the basis of Zacchi and Axelsson (1989). According to their research, a similar distillation column with a feed concentration of 15 % w/w requires energy of 3000 kJ/kg ethanol. With increasing feed concentration the energy requirement decreases; thus, for the given feed composition of 28 % w/w ethanol, we extrapolated the energy demand to approximately 2000 kJ/kg.

3.2 Response surface contour plots of energy demand

Although the regression model gives us a handle on the behavior of the energy consumption, the graphical representation of the fitted response surface is much easier to interpret. A contour plot is a

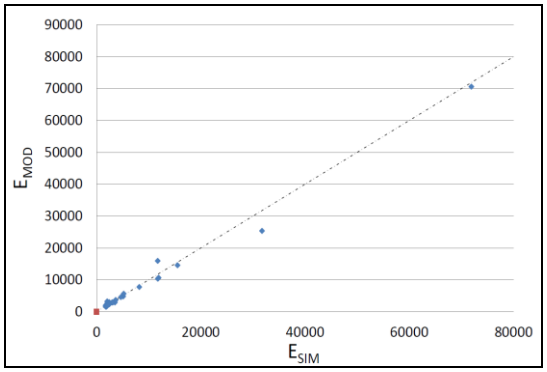

Figure 2: Predicted energy values by the regression model, E_{MOD}, versus simulated Aspen Plus energy values, E_{SIM}, 45 ° line added

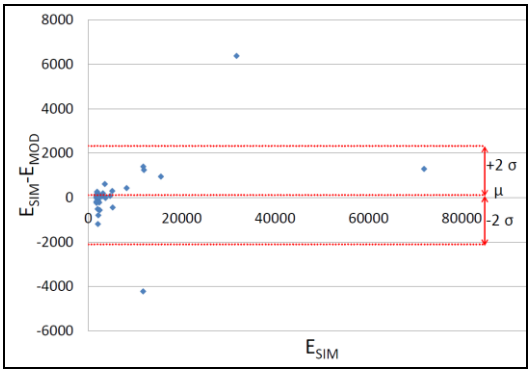

Figure 3: Residuals (error E_{SIM}-E_{MOD}) versus simulated Aspen Plus energy values, E_{SIM} and statistical confidence interval

convenient representation of the 3-dimensional response surface in a 2-dimensional plot. As there are only 2 axes in the plot, one out of three variables has to be kept constant.

Given the earlier derived threshold of 2000 kJ/kg energy demand, the corresponding contour line borders a region of lower energy demand. For example, Figure 4a gives the region of low energy demand for a constant number of stages (N=10), but varying influence of FSL and CT. The full circles at the margins and in the middle of the plot denote the position of the designed experiments with 5 levels. The variable space is rather sparsely covered by experiments, but still facilitates a powerful process analysis and interpretation. The contour plots shown in Figure 4a-f help to find process conditions, for which the energy requirement of the distillation column is below 2000 kJ/kg. The following in-detail interpretation of the graphical results is very well in accordance with fundamental distillation theory.

3.2.1 Lowest versus highest number of column stages

For the smallest of the investigated distillation columns that has N = 10 stages, the highest possible top-concentration of ethanol is 78.8 % w/w, but only if the feed stage is located in the middle column region (FSL = 55 %). More flexibility on the feed stage location, that is, feeding closer to the top stage, is possible if a lower top concentration (CT = 75–78 % w/w) is acceptable within the process. (Figure 4a).

The largest distillation column with N=26 stages has better energy efficiency, which is clearly reflected in Figure 4b. The process conditions can be varied in a large range without exceeding the limit of 2000 kJ/kg energy consumption. Nevertheless, if the goal is to reach the highest ethanol concentration (CT = 90 % w/w), the feed must be added between stage 14 and 18, counted from the top (FSL = 55–70 %). The energy demand would increase, if the feed is placed closer to the column condenser.

3.2.2 Lowest versus highest ethanol concentration

In Figure 4c and 4d the ethanol concentration at the top is kept constant at 75 and 90 % w/w, respectively. The general principle is that a higher ethanol concentration at the column top needs more energy in the distillation. This principle is clearly reflected, because the region of low energy demand is very limited for the highest ethanol concentration in Figure 4d. The advice derived from this contour plot is to use a distillation column with 20 or more stages, and locate the feed entrance close to the bottom of the column (FSL = 60-75 %). In the case of low ethanol concentration (CT = 75 %), a low energy consumption is fulfilled in a wide range of parameters setup (Figure 4c). Some restrictions on the feed stage location are found for smaller columns, in which the feed should not be placed too close to the condenser or reboiler, but preferably in middle of the column.

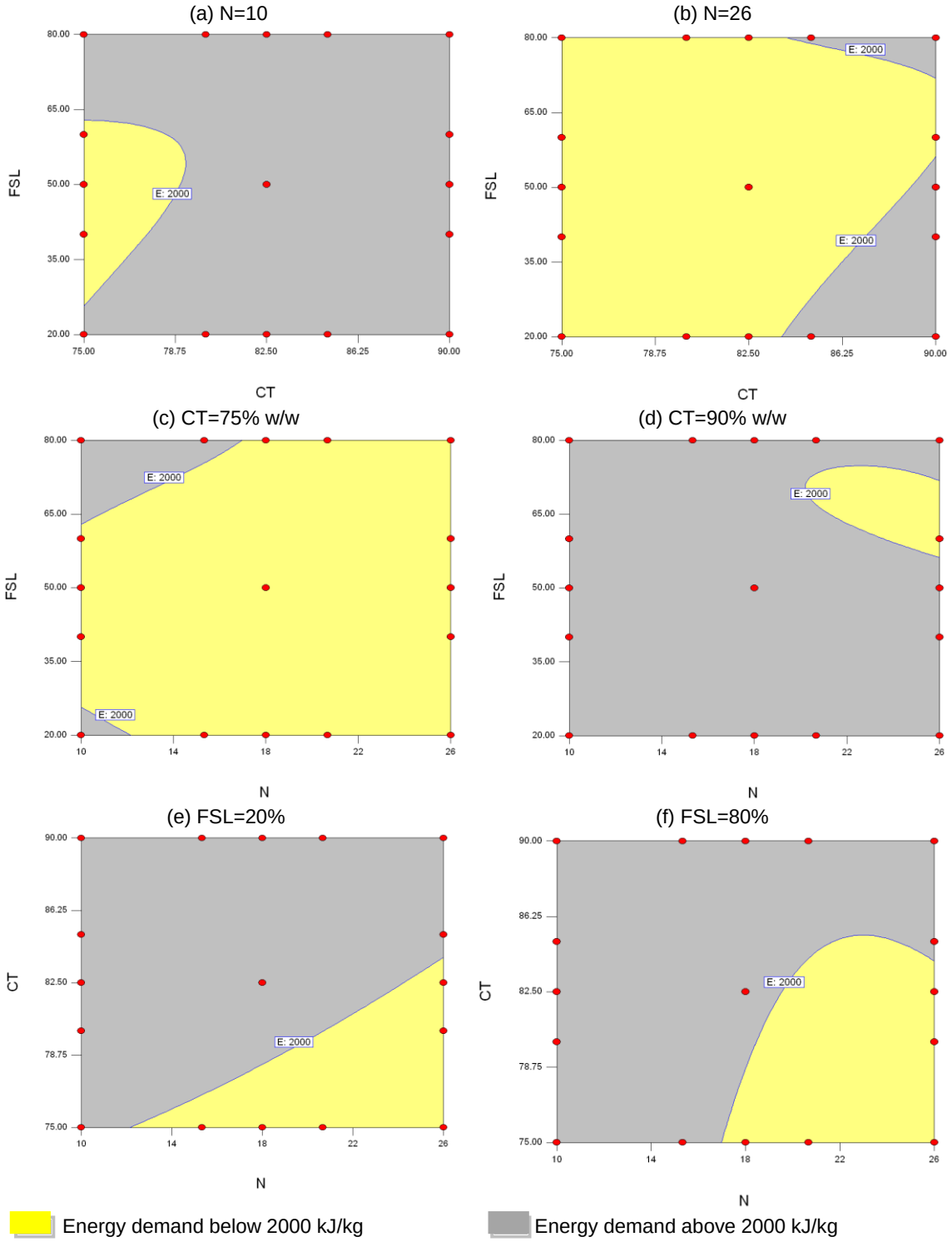

Figure 4: 2D contour plots with contour line at 2000 kJ/kg energy demand and one out of three column variables fixed. The region separated by the contour line indicates minimum energy conditions for the distillation column

3.2.3 Highest versus lowest feed stage location

In Figure 4e and 4f the feed stage location, FSL, is fixed to the minimum (20 %) and maximum values (80 %). If the feed enters at the top of the column, i.e. close to the condenser (FSL = 20 %), the required low energy consumption is only fulfilled for larger columns and lower ethanol concentration at the top (Figure 4e). If the feed enters very close to the bottom where the reboiler is situated, it is recommended to use a column with at least 18 stages. The maximum possible ethanol concentration is 82 % w/w, which can be reached in a column with 23 stages.

3.3 Model optimization for minimum energy demand

Optimization of the regression model given in Eq. (1) means to find the best values for the three column variables N, FSL, and CT that minimize the energy demand of distillation. The mathematical optimization with boundary conditions (see Section 2.1) is performed in Design Expert. For the investigated distillation system, the energy consumption can be minimized to $E_{MOD,MIN}$=1573 kJ/kg. To reach this minimum value, the recommended distillation column should have N=20 stages, the feed should be located in the middle of the column on stage number 10 (FSL=50 %), and the product at the column top should contain CT=78 % w/w ethanol.

The plausibility of the predicted minimum energy conditions (N=20, FSL=50, CT=78) is checked by a final Aspen Plus simulation run. The simulated value is E_{SIM}=1817 kJ/kg, and differs from the modeled minimum value, $E_{MOD,MIN}$ by only 244 kJ/kg or 13 %. It should be noted that the energy minimum $E_{MOD,MIN}$, predicted by the regression model, cannot be reached in Aspen Plus in any conditions.

4. Conclusions

Design of experiments (DoE) offers powerful tools (1) to plan experiments by D-optimal experimental design, (2) to describe the underlying process by the Response Surface Methodology, RSM, (3) to quantify the relationship between variables and the response by an empirical regression model, and (4) to optimize the process for a given response by minimizing the regression model.

Mathematical model clearly describes the distillation column with the least amount of simulation effort possible. However, it is an empirical model, based on process simulations, without experimental proof. While the statistical error of the model can be quantified, the simulation error of Aspen Plus compared to a real distillation column is unknown. In spite of the fact that the model is not perfect, the statements derived from it are very well in accordance with the principles known from distillation theory.

In contrast to trial and error searches of univariate experiments, we have a model that can be mathematically optimized. The search for optimum, that is minimum energy consumption, can be based on the solid ground of a well-described process. An additional advantage is that once the low energy regions are known, they can be combined with cost investigations. Eventually, the trade-off between energy demand, investment costs, and operational costs is reached much faster and in a more reliable way applying the investigated method.

References

Bas D., Boyaci I. H., 2007, Modeling and optimization I: Usability of response surface methodology. Journal of Food Engineering, 78, 836-845.

Fazlali A., Hosseini S., Yasini B., Moghadassi A., 2009, Optimization of operating conditions of distillation columns: an energy saving option in refinery industry. Songklanakarin Journal of Science and Technology, 31, 661-664.

Jacquez J. A., 1998, Design of Experiments. Journal of the Franklin Institute, 335B, 259-279.

Wang H., Cui X., Wang R., Li C., 2012, Response surface optimization of the operating parameters for a complex distillation column based on process simulation. Energy Procedia, 16, 571 – 578.

Wang H., Li C., 2010, RSM optimization of the operating parameters for a buthanol distillation column. Asia- Pacific Journal of Chemical Engineering, 7, 117–123.

Zacchi G., Axelsson A., 1989, Economic evaluation of preconcentration in production of ethanol from dilute sugar solutions. Biotechnology and Bioengineering, 34, 223-233.