Optimization of methanol production via CO$_2$ hydrogenation: comparison of sampling techniques for process modeling

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Abstract

This paper compares performances of three different sampling techniques in representing nonlinear response surface for optimization problem, with five decision variables. Three studied sampling techniques are Central Composite Design (CCD), Box–Behnken Design (BBD) and Latin Hypercube Sampling (LHS). Simulation – based optimization of methanol production via CO$_2$ hydrogenation process was used as a case study. The objective of this optimization problem is to minimize the methanol production cost. The nonlinear model (the objective function) represents the relationship between operating conditions and methanol production cost. The results show that the response surface of this case study is trend to be the third order model (with $R^2$ value greater than 0.97) for all sampling techniques. BBD is the most suitable sampling technique in this study, resulting in the percent error less than 1.2. The LHS shows comparable performance with BBD if it has suitable number of sample points.

1. Introduction

Carbon dioxide (CO$_2$) is the major species of greenhouse gas that causes global warming. Large quantities of CO$_2$ mostly release from fossil-fuel-fired power plants. One of studies in controlling the amount of CO$_2$ releases to the atmosphere is to convert CO$_2$ to valuable products such as methanol. Generally, methanol can be produced from synthesis gas, and CO$_2$ hydrogenation. A few works on simulation – optimization of methanol synthesis were studies.

For methanol synthesis from synthesis gas, the effect of changes in operating conditions on the production rate was studied to maximize the production rate [1]. The studied operating parameters were feed flow rate, pressure and temperature of feed, and the cooling water temperature. The results showed that the methanol production rate can be increased by 7% with higher feed pressure and lower feed temperature. For the study of methanol synthesis via CO$_2$ hydrogenation, Grazia Leonzio [2] developed mathematical model of the reactor used in methanol production. CCD was used as sampling technique. The impacts of reaction temperature, reaction pressure, H$_2$/CO$_2$ ratio, and the recycle factor on methanol production rate and reactor volume were studied. The performances of methanol production via synthesis gas and CO$_2$ hydrogenation were compared under constant reaction temperature of 245 °C [3]. The results showed that the process of methanol production via CO$_2$ hydrogenation consumed more utilities than the process of methanol production via synthesis gas.

One of important factors in process optimization is choosing the sampling technique used in constructing the test matrix for collecting the data. The obtained data is then used to represent the surface

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of the objective function. A few works were studied on comparison of sampling techniques in terms of their performances and required number of sample points. When comparing among full factorial design (FFD), CCD, and Doehlert design in modelling of the microbial growth rate, the CCD and FFD were found to be the most suitable techniques for this application [4]. By considering CCD, FFD, and BBD, the BBD and CCD require less work and shorter time of experiment than FFD [5]. The CCD and three – level full factorial design were found to represent better model than BBD and two – level full factorial design [6]. When comparing between performances of FFD and BBD used in the response surface methodology, BBD was recommended for the optimization due to it requires a smaller number of experimental runs [7]. A comparison of BBD and CCRD (central composite rotatable design) for three variables was studied in cosmeceutical application to identify prediction accuracy of the mathematical model. The predicted value using CCRD was found to be closer to the actual value than the one obtained from BBD [8]. The quadratic regression models constructed from BBD and CCD were validated in the study of CO₂ fixation. Both BBD and CCD showed good agreement between model predictions and experimental data [8].

However, comparison of BBD, CCD and Latin Hypercube sampling (LHS) performances in representing the nonlinear model for response surface methodology with large number of decision variables (more than three variables) has not been studied. This paper compares the performances of three different sampling techniques, which are BBD, CCD, and LHS, in representing the nonlinear model with five decision variables. Since the methanol synthesis via CO₂ hydrogenation consumes high utilities, the optimization of methanol synthesis from CO₂ hydrogenation was used as case study. The objective is to minimize methanol production cost per ton-produced methanol.

This paper is organized as follows: Section 2 gives details of process simulation and economic evaluation. Section 3 provides simulation – based optimization methodology used in this work including details of each studied sampling technique. Section 4 discusses the performances of each sampling techniques on the optimum solutions, and prediction accuracy of the obtained nonlinear model. Section 5 provides the conclusions.

2. Process simulation and economic evaluation
This paper studies comparison of sampling techniques used in data collection to construct the nonlinear models in term of their performance. The considering nonlinear models are the second and third order models. The simulation – based optimization of methanol synthesis from CO₂ hydrogenation process using response surface methodology was used as case study. The objective is to minimize the methanol production cost per ton-produced methanol. In what follows, we explain the details of the process simulation and economic evaluation.

2.1 Process simulation
The process flow diagram of methanol production via CO₂ hydrogenation is shown in figure 1. The process was simulated using Aspen Hysys version 10. Peng – Robinson was used as thermodynamics package. In the process, 1,000 kmole per hour of carbon dioxide and 3,000 kmole per hour of hydrogen at conditions of 40 °C and 20 bar was mixed, compressed, and heated up to the reaction temperature. The stream was then sent to the first equilibrium reactor, which the reactants were partially converted to methanol [9]. A separator separated the liquid methanol product while the unreacted CO₂ and H₂ reactants enter the second equilibrium reactor to produce more methanol product. The pressure of gas phase stream leaving the second reactor was reduced to recover methanol product as liquid phase. The combined liquid methanol product was sent to the first distillation column to separate the light
components (CO, CO$_2$, and H$_2$) at the top of the column. The bottom product, which are mixture of methanol and water, was sent to the second distillation column. The methanol was then distillated as the product at the top of the column with purity of 99.5% by mole.

![Simulation of methanol production via CO$_2$ hydrogenation process.](image)

**Figure 1.** Simulation of methanol production via CO$_2$ hydrogenation process.

2.2 Economic evaluation

Both capital and operating cost were included in calculation of methanol production cost. The capital cost involves all major equipment, except pump and piping. The capital cost was estimated using equations and data from CAPCOST program [10]. The data was adjusted for inflation from year 2001 (CEPCI value of 297) to 2017 (CEPCI value of 541.7) [11]. All assumption used in the economic analysis are the same as work of [9].

3. Optimization Methodology

This section describes simulation – based optimization algorithm and detail of each studied sampling technique. For methanol production via CO$_2$ hydrogenation process, the sensitivity analysis results from the work of [9] showed that there were five parameters that show significant impacts on the methanol production cost. Five parameters are inlet pressure to the first reactor, inlet temperature to the first distillation column, inlet temperature to the first reactor, inlet pressure to the second reactor, and outlet temperature of the liquid stream cooler after the second reactor. These five parameters were studied as decision variables for the optimization problem in this work.

Figure 2 represents simulation – based optimization algorithm. In the algorithm, the range of each decision variables was first determined. Table 1 shows the ranges of decision variables studied in this work. Then, $2^k$ factorial design is used to construct a test matrix of the decision variables. Then, process simulation, coupled with cost analysis, is run corresponding to operating conditions in the test matrix to obtained methanol production cost. The data of operating conditions and the corresponding methanol production costs are then combined to fit the first order model by regression analysis (using Design Expert 11 software). If the data fits the first order model ($R^2$ greater than 0.7), steepest descent [12] is performed to move operating conditions to the region of lower methanol production cost. If the data does not fit the first order model, it means that the data are in nonlinear region. The sampling technique (BBD or CCD or LHS, Section 3.1) is performed to collect more data in nonlinear region. The data of operating conditions from the sampling technique and the corresponding methanol production cost from the process simulation are combined to fit the nonlinear model (the second or third order
model). The nonlinear model is then used as the objective function in optimization problem. Microsoft Excel Solver is used to solve the optimization problem for the minimum methanol production cost.

![Diagram](image)

**Figure 2.** Simulation-based Optimization algorithm.

| Decision variables                        | Range          |
|-------------------------------------------|----------------|
| 1. Inlet pressure to the 1st reactor (bar)| 56.0 – 58.0    |
| 2. Inlet temperature to the 1st reactor (°C)| 192.0 – 196.0 |
| 3. Inlet pressure to the 2nd reactor (bar)| 110.0 – 118.0 |
| 4. Outlet temperature of the liquid stream cooler after the 2nd reactor (°C)| 74.5 – 78.5 |
| 5. Inlet temperature to the 1st distillation column (°C) | 48.0 – 50.0 |

**Table 1.** Ranges of decision variables.

### 3.1 Sampling techniques

Three different sampling techniques were used to construct the test matrix of data for constructing the nonlinear model. The studied sampling techniques are Central Composite Design, Box-Behnken Design, and Latin Hypercube Sampling.

**3.1.1. Central Composite Design (CCD).**

The design consists of three parts: full factorial design, an additional design of axial point, distance $\alpha$ from its center point and a center point, $\alpha$-values depend on the number of variables and can be calculated by $\alpha = (2^k)^{1/4}$, where $k$ is the number of independent variables [12]. The required number of sample points can be calculated by $N = 2^k + 2k + C_p$, where $C_p$ is the number of replications of a center point. Figure 3A represents code level of data set obtained from CCD with three decision variables.
3.1.2. Box-Behnken Design (BBD).
This sampling technique is three-level factorial arrangement, which consists of data points from $2^k$ factorial design, an overall center point and a center point at each side. This sampling technique is more efficient for the first- and second-order mathematical model [12]. The required sample points of this technique can be calculated by $N = 2k (k – 1) + C_p$ where $k$ is the number of independent variables, and $C_p$ is the number of replications of a center point. Figure 3B represents code level of data set obtained from BBD with three decision variables and a center point.

3.1.3. Latin Hypercube Sampling (LHS).
The number of data points ($N$) of this sampling technique is arbitrary. The test matrix is constructed by equally dividing the range of 0 (zero) to 1 (one) into $N$ ranges. Then, a sample points is randomly picked from each range of each variable. After that, sample points of each independent variable are randomly matched to the ones of other variables. Figure 3C represents code level of data set obtained from LHS with two decision variables and five sample points.

![Box-Behnken Design Diagram](image)

**Figure 3.** Code variables of different sampling techniques: A: CCD with three decision variables, B: BBD with three decision variables, C: LHS with two decision variables and five sample points.

3.2. Optimization formulation
In this work, the objective function is the nonlinear model that represents the relationship between the operating parameters (decision variables) and the methanol production cost. Four different nonlinear models were studied as the objective function. The four models are a full second – order model, a second – order model with only significant parameters, a full third – order model, a third – order model with only significant parameters.

The form of a full second – order model is shown in equation (1).

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_{i}^2 + \sum_{i<j} \beta_{ij} x_i x_j$$

(1)

The form of a full third – order model is shown in equation (2).


\begin{equation}
 y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \sum_{j=1}^{i-1} \beta_{ij} x_i x_j + \sum_{i=1}^{k} \sum_{j<i} \beta_{ij} x_i^2 + \sum_{i<j<k} \beta_{ijk} x_i x_j x_k
\end{equation}

The constraints depend on the sampling technique used in constructing the nonlinear model. The optimization formulation used in this work are shown below. The code variables of each decision variable are used in fitting the nonlinear model, so all the constraints are in code variables.

\text{MIN:} \quad \text{nonlinear model(four different models)}

\text{Decision variables:} \quad x_i \quad ; \quad i = 1, 2, 3, 4, 5

\text{Subject to:} \quad -1 \leq x_i \leq 1 \quad \text{for BBD and LHS}

\quad -2.37 \leq x_i \leq 2.37 \quad \text{for CCD}

4. Results and discussion

4.1 Steepest descent

The steepest descent was performed to search for the region of lower methanol production cost. The steepest descent moves the operating conditions to the new middle value of decision variable ranges as follows: inlet pressure to the first reactor: 58.84 bar, inlet temperature to the first reactor: 184.0 °C, inlet pressure to the second reactor: 106.64 bar, outlet temperature of the liquid stream cooler after the second reactor: 65.54 °C, and inlet temperature to the first distillation column 52.75 °C. All these new operating conditions were then used as the middle values of ranges of decision variables. The step size of each decision variable is the same as the initial data set. Theses ranges of operating conditions coupled with experimental designs were then used in data collection for constructing nonlinear model.

4.2 Optimal conditions

Three different sampling techniques which are Central Composite Design (CCD), Box - Behnken Design (BBD), and Latin Hypercube Sampling (LHS) were studied. These three sampling techniques were used for choosing data set to represent the nonlinear model (objective function in optimization problem). Since the required sample points for LHS is arbitrary, three different number of sample points (12, 41, and 80) were studied for LHS. Therefore, there five case studies of sampling technique in this work: 1.) BBD, 2.) CCD, 3.) LHS with 12 sample points, 4.) LHS with 41 sample points, 5.) LHS with 80 sample points.

To study abilities of sampling techniques in covering specific regions in response surface, two different nonlinear model were studied. The two nonlinear models are second – order and third – order models. For both nonlinear models, the full model and the model contains only significant parameters (with P – value < 0.05) were studied. Five operating parameters studied in this work are inlet pressure to the first reactor, inlet temperature to the first reactor, inlet pressure to the second reactor, outlet temperature of the liquid stream cooler after the second reactor, and inlet temperature to the first distillation column.

4.2.1 The model fitting with second – order model.

Tables 2(a) and 2(b) show the optimum operating condition obtained from the second – order models constructing with five different case studies. Table 2(a) shows the results of response surface function with full second – order model while Table 2(b) shows the results of the model contains only significant parameters. From both Tables 2(a) and 2(b), the results show that the obtained optimum operating conditions are in similar region for all five case studies. The optimum production cost of methanol is
around $566 with the optimum operating conditions as follows: inlet pressure to the first reactor: 56 – 60 bar; inlet temperature to the first reactor: 184 – 186 °C; inlet pressure to the second reactor: 106 – 107 bar; outlet temperature of the liquid stream cooler after the second reactor: 65– 66 °C, and inlet temperature to the first distillation column 53 °C.

When we consider the values of R – squared and adjusted R – squared for both full model and model contains only significant terms, the values are in between 0.7 – 0.9. This means the second – order model is not well suited for the data.

**Table 2(a).** The optimum conditions obtained from the 2nd order model.

| Number of sample points          | Sampling Techniques |
|----------------------------------|---------------------|
|                                  | BBD  | CCD  | LHS  | LHS  | LHS  |
| R-squared                        | 0.8764 | 0.713 | 0.9217 | 0.8438 | 0.7789 |
| Adjusted R-squared               | 0.8297 | 0.4521 | 0.8565 | 0.7849 | 0.7308 |
| Inlet pressure to the 1st reactor (bar) | 57.8  | 56.5  | 59.1  | 59.3  | 59.2  |
| Inlet temperature to the 1st reactor (°C) | 183.6 | 184.0 | 186.0 | 186.0 | 186.0 |
| Inlet pressure to the 2nd reactor (bar) | 106.3 | 106.1 | 106.4 | 106.5 | 106.3 |
| Outlet temperature of the liquid stream cooler after the 2nd reactor (°C) | 65.4  | 65.1  | 67.5  | 65.8  | 66.0  |
| Inlet temperature to the 1st distillation column (°C) | 52.8  | 52.8  | 52.9  | 52.8  | 52.7  |
| Predicted methanol production cost ($/ton) | 557.55 | 535.83 | 557.28 | 555.68 | 558.20 |
| Actual methanol production cost ($/ton) | 565.48 | 566.00 | 566.07 | 565.99 | 565.98 |
| % Error                          | 1.40  | 5.33  | 1.55  | 1.82  | 1.37  |

**Table 2(b).** The optimum conditions obtained from the 2nd order model with significant variables.

| Number of sample points          | Sampling Techniques |
|----------------------------------|---------------------|
|                                  | BBD  | CCD  | LHS  | LHS  | LHS  |
| R-squared                        | 0.8705 | 0.7127 | 0.9129 | 0.8371 | 0.7715 |
| Adjusted R-squared               | 0.8610 | 0.6824 | 0.9017 | 0.8199 | 0.7563 |
| Inlet pressure to the 1st reactor (bar) | 57.8  | 56.5  | 59.1  | 59.1  | 59.1  |
| Inlet temperature to the 1st reactor (°C) | 183.6 | 184.0 | 186.0 | 186.0 | 186.0 |
| Inlet pressure to the 2nd reactor (bar) | 106.6 | 106.6 | 106.5 | 106.6 | 106.6 |
| Outlet temperature of the liquid stream cooler after the 2nd reactor (°C) | 65.5  | 65.5  | 65.5  | 65.5  | 65.5  |
| Inlet temperature to the 1st distillation column (°C) | 52.7  | 52.7  | 52.7  | 52.7  | 52.7  |
| Predicted methanol production cost ($/ton) | 559.59 | 532.84 | 553.10 | 557.27 | 559.63 |
| Actual methanol production cost ($/ton) | 565.54 | 566.08 | 566.18 | 566.07 | 566.28 |
| % Error                          | 1.05  | 5.87  | 2.31  | 1.55  | 1.17  |
4.2.2 The model fitting with third – order model.

Tables 3(a) and 3(b) show the optimum operating condition obtained from the third - order models constructing with five different case studies. Table 3(a) shows the results of response surface function with full third – order model while table 3(b) shows the results of the third – order model that contains only significant parameters. From both Tables 3(a) and 3(b), the results show that the obtained optimum operating conditions are in similar region for all five case studies, which are the same as the results obtained from the second – order model.

When considering the values of R – squared and adjusted R – squared, the values are higher than the ones of the second – order model for all sampling techniques. This means that the objective function of this optimization is trend to be the third – order model.

| Table 3(a). The optimum conditions obtained from the 3rd order model. |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| Number of sample points | BBD | CCD | LHS | LHS |
|-----------------|---|---|---|---|
| R-squared | 0.9901 | 0.9743 | 0.9999 | 0.9952 | 0.9699 |
| Adjusted R-squared | 0.9687 | 0.8459 | 0.9999 | 0.9804 | 0.9410 |
| Inlet pressure to the 1st reactor (bar) | 59.8 | 58.8 | 59.8 | 59.8 | 59.0 |
| Inlet temperature to the 1st reactor (°C) | 185.2 | 186.3 | 186.0 | 186.0 | 186.0 |
| Inlet pressure to the 2nd reactor (bar) | 106.2 | 105.6 | 106.6 | 110.6 | 110.6 |
| Outlet temperature of the liquid stream cooler after the 2nd reactor (°C) | 65.5 | 65.4 | 65.5 | 65.8 | 65.1 |
| Inlet temperature to the 1st distillation column (°C) | 52.8 | 52.9 | 52.7 | 53.7 | 53.7 |
| Predicted methanol production cost ($/ton) | 559.37 | 510.11 | 435.46 | 519.29 | 552.19 |
| Actual methanol production cost ($/ton) | 564.71 | 566.39 | 566.07 | 566.34 | 566.00 |
| % Error | 0.95 | 9.94 | 23.07 | 8.31 | 2.44 |

| Table 3(b). The optimum conditions obtained from the 3rd order model with significant variables. |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| Number of sample points | BBD | CCD | LHS | LHS |
|-----------------|---|---|---|---|
| R-squared | 0.9901 | 0.9742 | 0.9975 | 0.9801 | 0.9598 |
| Adjusted R-squared | 0.9880 | 0.9690 | 0.9953 | 0.9725 | 0.9531 |
| Inlet pressure to the 1st reactor (bar) | 59.8 | 58.8 | 59.8 | 59.8 | 59.8 |
| Inlet temperature to the 1st reactor (°C) | 185.2 | 186.3 | 185.0 | 186.0 | 186.0 |
| Inlet pressure to the 2nd reactor (bar) | 106.2 | 106.6 | 110.6 | 106.5 | 106.6 |
| Outlet temperature of the liquid stream cooler after the 2nd reactor (°C) | 65.4 | 65.5 | 65.5 | 65.6 | 65.4 |
Inlet temperature to the 1st distillation column (°C) & 52.8 & 52.8 & 52.8 & 52.8 & 52.8 \\
Predicted methanol production cost ($/ton) & 559.35 & 512.18 & 546.73 & 555.84 & 562.31 \\
Actual methanol production cost ($/ton) & 565.65 & 566.52 & 566.46 & 566.07 & 566.06 \\
% Error & 1.11 & 9.59 & 3.48 & 1.81 & 0.66 \\
\hline

4.3 Prediction accuracy of the nonlinear models

The prediction accuracy of the nonlinear model was estimated by % error calculation of each model. The % error of the nonlinear model (row 10) was estimated by comparing the predicted methanol production cost (obtained from nonlinear optimization solver, row 8) with the actual production cost (row 9). The actual production cost was obtained by running the process simulation with the corresponding optimum operating condition.

For both studies of the second – order model (Tables 3(a) and 3(b)), the results show that CCD has the highest % error (5.3 - 5.8%), which means lowest prediction accuracy among all of five case studies. The nonlinear model constructed from LHS with 80 sample points has the lowest % error compared the others two case studies of LHS, which have a smaller number of sample points. The model constructed from BBD show comparable prediction accuracy with the model constructed from LHS with 80 sample points (with % error less than 1.5).

For the study of full third – order mode (Table 3(a)), the model constructed from BBD shows the highest prediction accuracy (with % error of 0.95). The model constructed from LHS with 80 sample points shows the second highest prediction accuracy. The model from LHS with 12 sample points yields the lowest accuracy (with % error of 23.07). This is because the third – order model has more interaction terms and more complicated than the second – order model, so it requires more sample points to be able to represent the specific highly nonlinear area. When only significant terms were used in the third – order model, the % error of the model constructing from LSH with 12 sample was dramatically decreased. This is because the model contains less mathematic terms than the full model, so less data points are required to represent the entire response surface. When we look at the CCD in both full model and the model with significant terms, the % errors are similar (around 9%). This means that the number of sample points is enough to represent the entire response area, but the sampling technique cannot cover some specific highly nonlinear region as it shows high % error.

5. Conclusion

The performances of three different sampling techniques in covering the nonlinear regions were compared. The simulation – optimization of methanol production process via CO₂ hydrogenation was used as a case study. The methanol production cost was minimized using response surface methodology. Two different nonlinear models (the second order and third order models) were used as the objective function. The results showed that the objective function of this case study is trend to be the third order model. For both full third – order model and the model contain only significant parameters, BBD shows the highest performance to represent these nonlinear models with % error less than 1.2. The nonlinear models constructed from CCD show the lowest prediction accuracy. By comparing LHS with different number of sample points, the nonlinear models constructed with 80 sample points shows high prediction accuracy while the ones constructed with 12 and 41 sample points show less prediction accuracy. The performance of LHS with 80 sample points is comparable to the BBD. Since the number of sample
points for LHS is arbitrary and has effect on its performance, determination of number of sample points needs to be further studied in order to effectively used LHS to represent the nonlinear region.

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