CHEMICAL ENGINEERING TRANSACTIONS Volume 24, 2011
Editor Sauro Pierucci

 ISBN 978-88-95608-15-0 ISSN 1974-9791
DOI: 10.3303/CET1124060

Towards the Assessment of Robustness in an Industrial Process System

Shyamal Gondkar[a], Edwin Zondervan[b*], Sivakumar Sreeramagiri [a], André B. de Haan[b]

[a]GE Global Research – Innovative Plastics Program, GE India Technology Centre Pvt. Ltd., Bangalore, India
[b] Eindhoven University of Technology
P.O. Box 513, 5600MB, Eindhoven, the Netherlands
e.zondervan@tue.nl

The scientific community has developed several tools in the last years that can be used to satisfy the economic- and environmental requirements of the design of a process system. However, tools for the systematic connection and evaluation between design and long- and short term operation is currently still at an infant stage. As design and operation are integrated, the overall complexity of such system increases significantly, ergo, the required computer power and capacities of solution algorithms also do.
In our work we develop a systematic framework to assess robustness and sustainability of a process design and the operational strategy of the design involving process intensification. We use a four-step methodology; first we define the objectives and develop suitable process models that describe the process intensification alternative, secondly we execute a response surface methodology to obtain better understanding of how to influence the objectives, thirdly we optimize and select the design alternatives and the operational strategies, and lastly we evaluate the design and operational alternatives.

1. Introduction

The chemical engineer is concerned with finding the best design and/or optimal operation of a chemical process plant. Often economic indicators determine whether or not a design and/or operation strategy is desirable. However, economic feasibility is no longer the only objective that needs to be satisfied; from environmental viewpoint the design and operation should also be sustainable. Also, in the last few years process intensification concepts have gained lot of attention. Process intensification is an integration effort, which combines classical chemical engineering with chemistry; materials and mechanical engineering design to miniaturize the process equipment, improve process efficiency and minimize environmental foot-print. The use of process simulators has become extremely popular for various purposes such as conceptual design, plant performance monitoring and plant optimization in the chemical process industries and refineries. The commercially available software tools allow users to build process models, and then run simulations without tedious calculations. However, tools

Please cite this article as: Gondkar S., Zondervan E., Sreeramagiri S. and De Haan A., 2011, Towards the assessment of robustness in industrial process system, Chemical Engineering Transactions, 24, 355-360
DOI: 10.3303/CET1124060

for the systematic connection and evaluation between design and long- and short term operation is currently still at an infant stage.

The design and operation of integration alternatives such as process intensification requires attention. For example, at the conceptual design stage, models are often used to make decisions or trade-offs on the process conditions and the design variables. In most of the modeling efforts, a real life process under investigation is represented by a simplified mathematical description. During each stage of the modeling effort there are uncertainties, which can influence the model response. In process engineering related problems three main types of uncertainties are encountered; structure uncertainty, input data uncertainty and uncertainty of the model parameters. The first one is due to the simplified mathematical form of a complex physical or chemical process. The second type is due to the lack or error in the measurements, fluctuations in the natural or process conditions etc. The third type may arise due to empiricism (empirical nature of the equations describing various parameters) or the error associated in the parameter regression of the model describing physical or chemical process. Because of the uncertainties associated with the modeling process the confidence intervals of the model need to be evaluated. Therefore, sensitivity analysis, which provides the contribution of the various uncertainties to the model output uncertainty, is performed to characterize the uncertainty associated with a model. Our initial objective is to provide an assessment framework for a process intensification alternative, which includes the best modeling approach with respect to its significance. Our ultimate objective is to use this methodology at a later stage to evaluate how economic (Total Annualized Costs) and emergy indicators (Lou et al., 2004) are influenced by operational variables and design variables.
This work includes a process intensification case-study, where heterogeneous catalytic reaction and separation are carried out in one fractional distillation column to simplify the process flow-sheet. We present the conceptual process model in Aspen Plus and use classical response surface methodology to obtain a simplified model. We also present an alternative to the classical response surface methodology, where the process model is directly integrated with the sampling and sensitivity analysis tool Crystal Ball and commercial meta-heuristic optimization software OptQuest.

2. Case-Study

In this study we looked at the possible processes for making bio-diesel. Biodiesel comprises of mixture of fatty acid esters. The traditional processes involving homogenous catalysts are considered to be highly energy intensive because of complex downstream processes which involves neutralization of the catalysts, recovery of excess alcohol, product isolation, purification steps for products and by-products, waste-treatment etc. Also there are other limitations on the feedstock for base-catalyzed reactions, for example, free fatty acid content resulting in the saponification and emulsion formation affecting product isolation. So the ideal process would involve a continuous flow reaction that does not deactivate or consume the catalyst and that further minimizes or eliminates the need for elaborate downstream separation and purification steps. The use of heterogeneous catalysts would result in simpler, cheaper

separation processes, a reduced water effluent load as well as lower capital and energy costs. In contrast, the main drawbacks are the requirement of more severe process conditions to speed-up the reaction rates, as well as the higher methanol to oil ratio needed for the transesterification reaction. Malero *et al.* (2009) have proposed a scheme for producing bio-diesel from the feedstock containing higher free fatty acid content containing a fixed bed heterogeneous catalytic reactor followed by couple of distillation columns to recover methanol, a decanter for separating crude fatty acid esters and crude glycerol and purification sections for fatty acid esters and glycerol.

3. Process Intensification Scheme

A scheme involving integrated heterogeneous catalytic reaction and distillation is modeled in Aspen Plus as shown in Figure-1, involving a distillation column containing heterogeneous catalyst. Oil feedstock is fed in the upper section whereas the methanol is fed few stages above the reboiler.

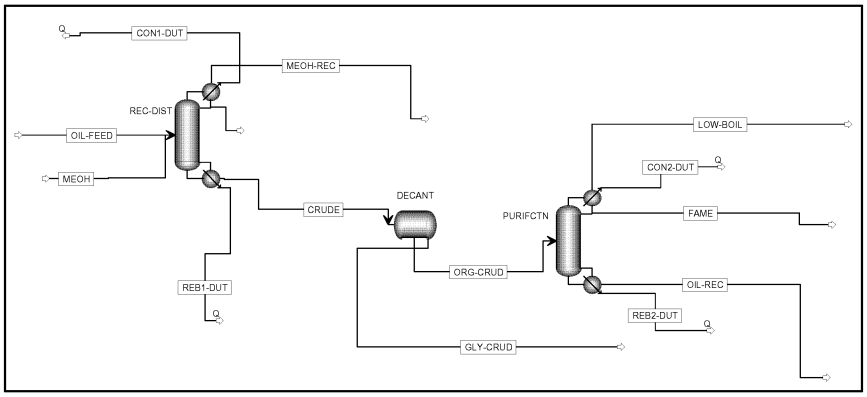

Figure 1: Aspen Plus flow-sheet indicating process intensification (integrated reaction and separation in a fractional distillation column) and further separation of fatty acid esters

The bottom product contains mainly fatty acid esters, unreacted feedstock, glycerol, water and methanol traces. The top product contains mainly methanol which can be recycled. The column was run under reflux conditions and column pressure was decided based on the temperature profile required inside the column in order to achieve desired reaction rates. The column conditions were decided such that water of reaction was discharged along with the column bottom product. Dortmund modified UNIFAC (Dortmund-UNIFAC) physical property model was implemented for this system. The reactions defined were mainly transesterification of triglycerides, esterification of fatty acids and hydrolysis of fatty acid esters. Simulations were run for various conditions to understand the column operations, as a part of screening strategy. The composition profile under one set of process conditions is shown in Figure-2. From this profile it can be seen that the water is concentrated in the bottom section of the column.

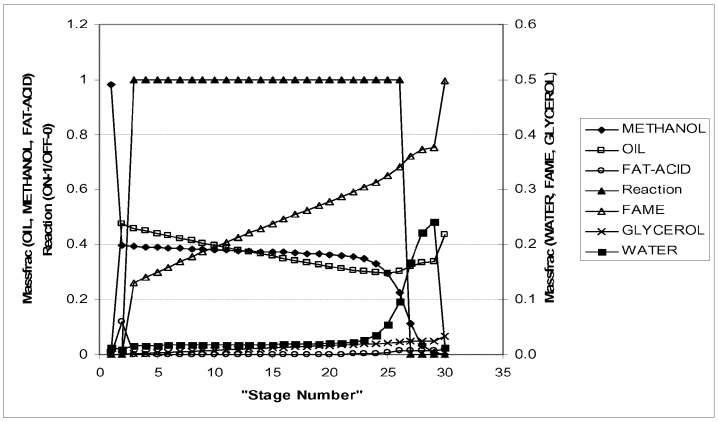

Figure 2: Steady state concentration profile of various species in the reactive distillation column simulated in Aspen Plus.

4. Design Methodology

Response surface analysis was carried out using classical design of experiments. For this purpose three factors were selected, namely, residence time on each stage, reflux ratio and methanol rate in the column feed. The responses obtained from Aspen Plus were production rate, methanol losses, energy consumption, column diameter and column height. A Centre composite design (CCD) was selected for the response surface analysis, which can be used to construct a second order model. Simulations were run at the design points to obtain the above responses. Uncertainties in the Aspen Plus model parameters and process conditions were not considered at this stage to obtain standard deviation of the responses at each design point. Using Design Expert-6 software tool data analysis (ANOVA) was performed for the mean values of the various responses. For heat duty and column diameter quadratic models were suggested, whereas for production rate, methanol losses and column height linear models were suggested. For all the responses except "methanol losses", the model statistics and diagnostic plots (normal probability plot of the studentized residuals to check for normality of residuals; studentized residuals versus predicted values to check for constant error; Box-Cox plot for power transformations) did not reveal any caution for further optimization exercise. For "methanol losses" the R^2 was estimated to be 0.63, which means higher error between the model prediction and the actual data, but Predicted R^2 of 0.49 and Adjusted R^2 of 0.6, allowed to use the model equation for further optimization exercise.

The optimization problem formulated as a single objective optimization problem by imposing various design requirements or constraints in order to test the framework. The point prediction for the optimized conditions was also carried out and results are given in Table 1. Using the above design strategy, the production rate of 523.5 kg/h was obtained. For some of the constraints like methanol losses or column height the model predictions were very close to the limit, and the confidence interval or prediction interval suggested that there was a high probability of violation of these constraints. Hence, in order to safeguard these design requirements there is a need of a factor of safety or a probabilistic design approach, which would be looked at a later stage.

We managed to simplify the process model using a classical DOE approach, but because of uncertainties (or error) associated with the model parameters the responses had a much larger confidence interval or prediction interval, which may result into over-design or violation of some of the constraints. To understand the capability of the existing scheme, we decided to couple the detailed process model directly to the sensitivity analysis tool and optimizer. For this purpose we selected Crystal Ball as sensitivity analysis tool and OptQuest (OptQuest for Crystal Ball 2000 V1.3) as meta heuristic optimization tool.

Table 1: Optimization results comparison, classical DOE vs. Integrated approach

Objective/Requirements	**Using Classical DOE (CCD)**								**Integrated**
	Point prediction	**SE Mean**	**95% CI low**	**95% CI high**	**SE Pred**	**95% PI low**	**95% PI high**		**Point prediction**
production rate (kg/h)	**524**	**21**	**478**	**569**	**46**	**422**	**625**		**630**
MeOH losses per unit product	**0.0489**	**0.0068**	**0.0342**	**0.0637**	**0.0213**	**0.0029**	**0.0950**		**0.0009**
Heat duty per kg product (Kcal) (for both the columns)	**-1020**	**14**	**-1051**	**-989**	**44**	**-1116**	**-924**		**-1019**
Column Diameter (m)	0.38	0.0078	0.36	0.40	0.0220	0.33	0.43		0.45
Column Height (m)	26.137	2.080	21.610	30.660	5.230	14.730	37.540		25.788
RD column distillate rate (kg/h)	87.0	5.2	75.4	98.5	12.5	59.5	114.5		125.9

The advantage of this approach is that the uncertainties around the Aspen Plus model parameters or process conditions may be specified in Crystal Ball, which would enable us to compute the variation in the responses and perform probabilistic optimization at a later stage. We also understand that this kind of an optimization would need lot more computational power, as it needs to run Aspen Plus simulation for each condition. In OptQuest we specified the identical design requirements as that of Design_Expert and defined production rate as an objective function to be maximized. After 1.000 trains no significant improvement in the objective function were observed, hence, the optimization was terminated. The final results of this optimization exercise are given in Table 1. A production rate of 630 kg/h was obtained, while most of the other requirements were similar or still lower than that obtained from classical DOE approach. This result shows that integrating the model with an optimizer leads to better performance. As the computational times are quite large there is still a need of a better methodology for assessment of a larger industrial system without compromising on the quality of the model.

5. Conclusion and future work

This paper presented a response surface methodology applied to the design of a process intensification scheme involving heterogeneous catalytic reaction and separation in a fractional distillation column. A classical centre composite design was directed to a simple model because of its usefulness in optimization of a large industrial system. To evaluate the performance of the response surface methodology, an improved optimization strategy was established which used commercial process simulation

software Aspen Plus, sensitivity analysis and sampling tool Crystal Ball and commercial meta-heuristic optimization software OptQuest, which lead to higher production rates, but also requires a lot of CPU resources, as for each scenario, Aspen simulations have to be executed.

Currently we try to circumvent the extensive Aspen simulation by meta-modeling techniques (See Dellino *et al.*, 2010), to deal with a highly non-linear design space or complex design. From literature we know that these meta-models result in faster analysis and reduced design cycle time.

References

Dellino, G., Kleijnen, J., and Meloni, C., 2010, Simulation-Optimization under Uncertainty through Metamodeling and Bootstrapping, Procedia Social and Behavioral Sciences, 2, 7640–7641.

Lou, H. H., Kulkarni, M. A, Singh, A.,and Hopper, J.R., 2004, Sustainability Assessment of Industrial Systems, Industrial and Engineering Chemistry Research, 43, 4233-4242.

Melero, J. A., Iglesias, J., and Morales, G., 2009, Heterogeneous acid catalysts for biodiesel production: current status and future challenges, Green Chem., 11, 1285–1308.