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Integration of Process Modeling with Laboratory Experiments in Conceptual Design: Bio-based Glycerol Dehydration Case Study

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Abstract

During the conceptual stage of process design, the experimental work to determine the reaction parameters is often carried out independently from the simulation work used to develop the conceptual process. The result of this disconnect is that the optimal process may remain undiscovered, since the process design engineer is constrained by the reaction conditions originally studied in the laboratory. By utilizing a methodology for integrating the process development with the laboratory experiments at the earliest stages, the process designer can ensure that the laboratory data is gathered only for economically viable and technically feasible process conditions. The methodology is illustrated through a case study on dehydration of bio-based glycerol.

Keywords: Conceptual Design, Glycerol Dehydration, Process Modeling

1. Introduction

In this work the development of a methodology for integrating process simulations with laboratory reaction experiments is presented using a case study example. The case study chosen to illustrate this proposed methodology is the conceptual design of a process for manufacturing an industrially important chemical product from sustainable, bio-based glycerol. The objective of this research is to identify cost effective processes with minimized environmental impacts that can utilize glycerol produced as a side product of biodiesel manufacturing. The environmental impacts are potentially significant, as current industrial production processes are based on crude oil derived feedstocks [1].

Using simulation models, the most economically viable and technically feasible process for further study in the laboratory can be identified. This contribution illustrates how process simulation tools are used in conjunction with experimental laboratory studies to develop an optimized process for switching production of an industrially important chemical from crude oil derived to sustainable, biomass derived feed stocks.

2. Background

The process investigated in this research is the catalytic dehydration of glycerol using an acid catalyst. Previously published literature on this reaction identified only the overall conversion and yield of the primary product [2]. These results have been published for both high pressure liquid and low pressure vapor phase reaction systems, however, the identity and yield of the side products were not reported. Therefore, significant additional work remains to be done to develop the parameters for this system of reactions. This contribution will illustrate how process simulations are used to guide the design and operation of a lab scale mini-plant to carry out the required reaction experiments. Furthermore, it will be demonstrated how the laboratory results are used to update the process simulation models to complete the process optimization. This approach not only ensures that the processes developed for the separation and purification of the product are based on economically optimized targets, but also minimizes the required laboratory work, since the experimental parameters are always based on viable and feasible operational boundaries. This serves to minimize the expenditure of both time and money for process development.

3. Methodology Development

By integrating process simulation tools with laboratory experiments, the development of an optimized industrial process can be streamlined. The methodology for process development is outlined as a flowchart (see Figure 1).

From this flowchart, it can be seen that process simulations are integrated in conjunction with laboratory experiments to ensure that the results are always based on economically viable and technically feasible conditions. In this way, the process simulations are used to direct the laboratory work, thus eliminating time spent on parameters that do not lead to optimized solutions. This can also potentially reduce the total expenditure of time and money in the laboratory due to the effect of streamlining the research and development process.

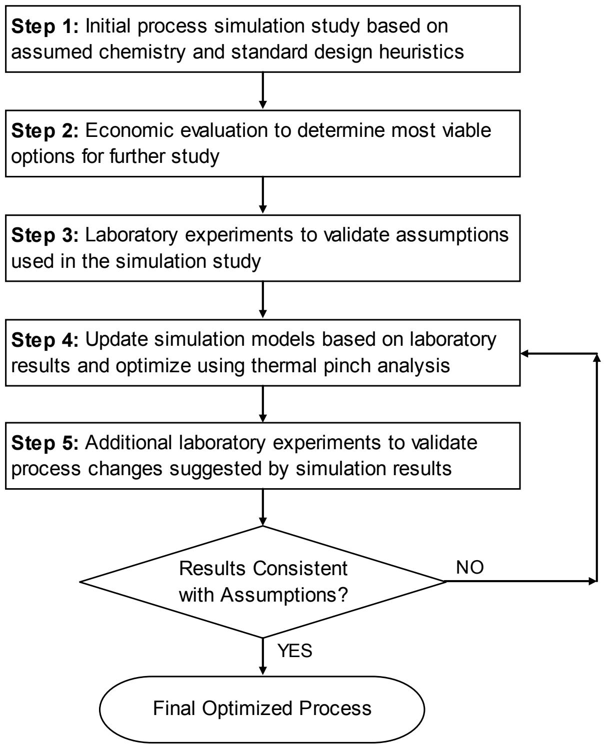

Figure 1. Flowchart for integrating process simulation work with experiments.

4. Case Study Example

The case study presented below is based on the application of the proposed methodology to an actual process development project. Through the case study, each step in the proposed methodology will be illustrated.

Methodology Step 1

The first step in the application of the methodology is the development of process simulation models based on an assumed reaction mechanism derived from literature data. Since two potential processes had previously been described – one based on reaction under high pressure liquid phase conditions and one based on low pressure gas phase conditions – this formed the basis for the initial process simulation studies [2]. A process model was developed for both the liquid phase and vapor phase processes including various recycle options using the Aspen Engineering Suite [3].

Methodology Step 2

The second step of the proposed methodology is the economic evaluation of these simulation options. The result of this analysis is described in previously published work on this research [4]. Since these results indicated that the vapor phase process held the best chance for economic viability, the laboratory experiments were based on this process.

Methodology Step 3

The third step is the validation of the simulation assumptions. The equipment used for the laboratory experiments is designed to model the vaporizer and packed bed reactor of the vapor phase glycerol dehydration process (see Figure 2). The product leaving the reactor is sampled via sample port QE, and analyzed using a gas chromatograph. With this equipment, the primary side products were identified and the overall conversion and yield were measured.

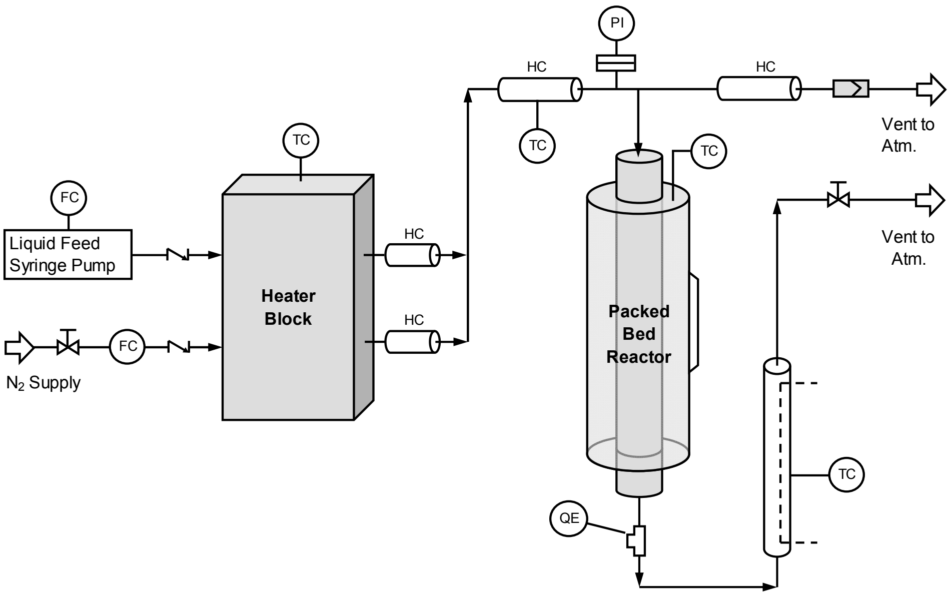

Figure 2. Schematic of laboratory equipment used in case study example.

Methodology Step 4

Step 4 of the methodology is to update the process simulation model with the experimental data to enable more detailed analysis. Use of a process simulation model allows more in-depth analysis of each component of the proposed industrial process. The results of this study indicated that the vapor phase process, although more economically attractive than the liquid phase process, was, for the conditions considered in this study, still not competitive with the industry standard process based on using crude oil derived raw materials [1].

The analysis of each component of the process indicated that the primary cause of the higher cost of the vapor phase glycerol process relative to this standard process is the contribution of the cost of energy per kilogram of product. Further analysis identified that the energy associated with vaporizing the feed was the largest contributor to the utility cost. This is due to the fact that the glycerol feed must be highly diluted in order to prevent the glycerol from reacting with itself. An engineering solution to avoid the problem of spending large amounts of energy vaporizing the diluent water must be found to make this glycerol dehydration process economically competitive.

As a result of the insights learned from the revised simulation model based on laboratory data, a new process has been developed to address the problem of vaporizing the water required for the vapor phase process. This new process has been modeled using the Aspen Engineering Suite to determine its economic viability. The results of this economic analysis are indicated in Figure 3.

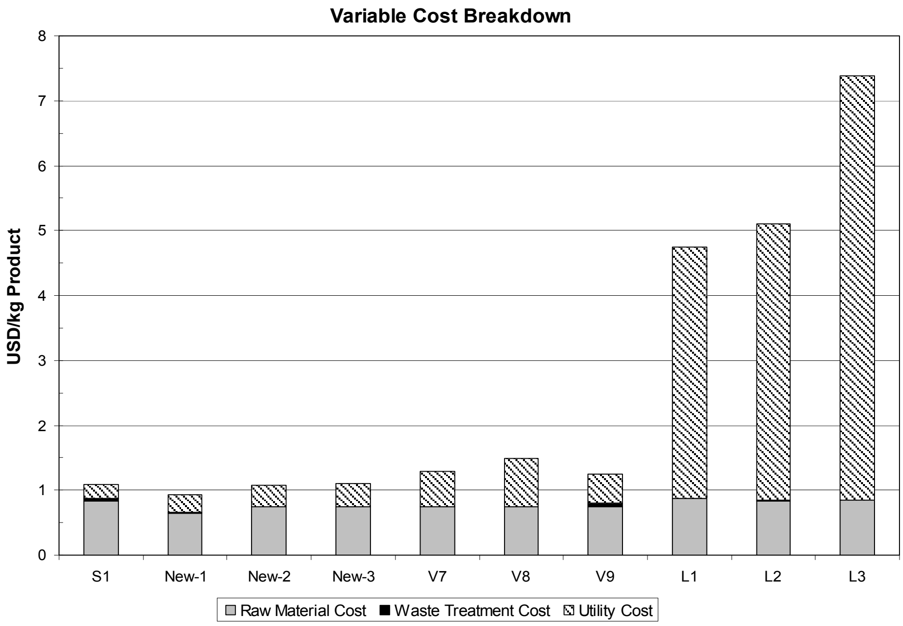

Figure 3. Economic analysis results from revised process simulation study.

The best case liquid phase (L1, L2, L3) and vapor phase (V7,V8,V9) process results and the standard case (S1) from the initial economic analysis [4] are included for comparison with the new process and its associated options (New-1, 2, 3). Since this new process is based on assumptions, the next step is to return to the laboratory to ensure that the assumptions made regarding glycerol conversion and product yield are valid.

Methodology Step 5

Step 5 of the methodology is then to determine if the laboratory results of the experiments using the new process conditions are consistent with the simulation results. If not, the process simulations will be modified again, followed by a return to the laboratory for further validation. Preliminary laboratory results using the new process conditions have been encouraging.

5. Results and Conclusions

In conclusion, it can be seen that by integrating the laboratory experiments with the process simulations, innovative process designs can be discovered. By applying the proposed methodology to this process development case study, an economically viable process has been developed. From the results presented, it is clear that without integrating process simulations with the laboratory experiments, the more economically attractive process conditions may not have been discovered. In fact, the initial poor economic results of the vapor phase process may have even led to the entire project being scrapped. Although additional work remains to be done to complete the process development, it is clear that the glycerol dehydration process now shows economic potential and warrants further study.

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