
# Experimental and numerical strategy for computer-aided identification of optimal intensified reactors and operating conditions

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***Highlights***

- A systematic modeling method is introduced in the iterative experimental strategy.
- A general PIS applied to chemical synthesis in liquid phase is developed.
- A case study, ethanolysis of vegetable oil by homogeneous base catalyst, is presented.

## 1. Introduction

Since a few years, intensified reactors, such as plug-flow microreactors, are alternatives to stirred tank reactors. Thanks to their high heat and mass-transfer rates, a better control of operating conditions makes it possible to reach a better product quality. However, it is difficult to predict quantitatively the real interest of intensified reactors without a large amount of experimental work. Hence, an iterative strategy[1] based on the combination of experimental design and modeling has been successfully developed to select the best reactor technology and corresponding optimal operating conditions while minimizing experimentation steps.

The objective of this work is to improve the iterative strategy[1] and to develop a process intensification software (PIS) in which the users can optimize their experimental strategy. In order to test the acceptance of the strategy and the software, two chemical syntheses concerned with valorization of vegetable oil (ethanolysis of vegetable oil by homogeneous base catalyst[2] and epoxidation of vegetable oil by percarboxylic acid[3]) will be achieved.

## 2. Methods

Figure 1 shows the new algorithmic scheme of strategy.

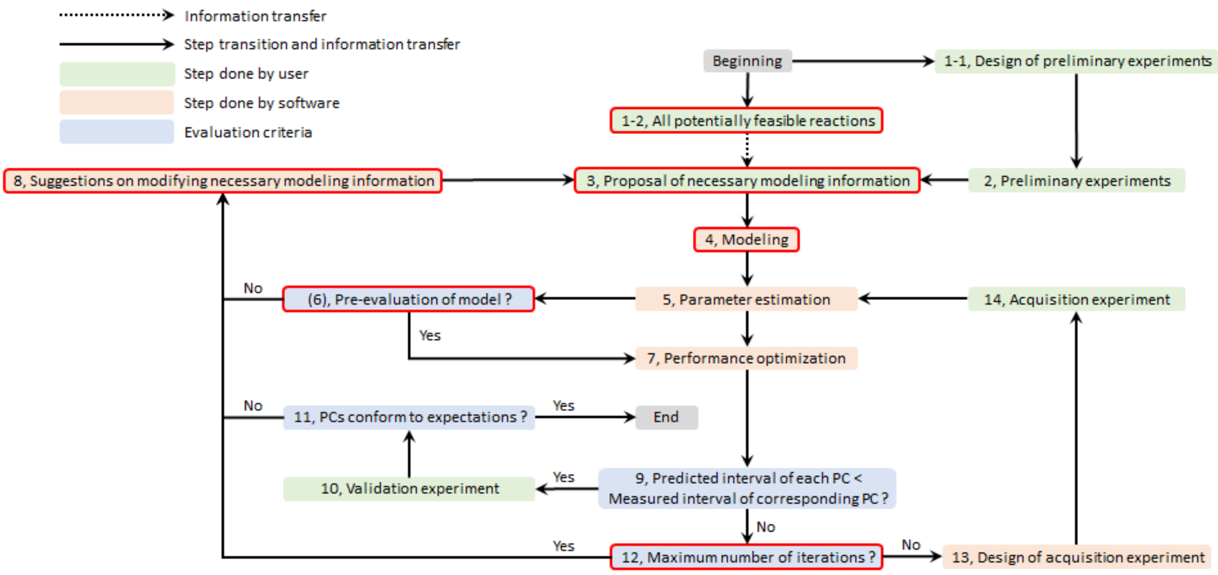


**Figure 1.** Algorithmic schema of strategy (PC: performance criterion)


Compared with the previous iterative strategy[1], the steps framed in red have been improved:

- Improvement 1: To enlarge the applicability of the strategy, after selection of the involved reactions and the limiting phenomena, a systematic modeling method is introduced.
- Improvement 2: To avoid unnecessary work, for a new model, before first performance optimization, the new model is pre-evaluated to check if all measured quantities are coherent with those simulated.
- Improvement 3: Before designing the acquisition experiment, it is necessary to check if the maximum number of iterations is reached.
- Improvement 4: Following a failure, some suggestions on modifying the necessary information for modeling will be proposed from the current experimental data[4], and then the user will be able to select one proposal among those recommended.

## 3. Results and discussion

The initial version of PIS consisting of four functions (process modeling, parameter estimation, process optimization, design of experiments) has been developed by using MATLAB. Its functionalities have been tested successfully with a simple and confidential numerical project. This work presents application of the methodology and software tool to an experimental complex case study: the ethanolysis of vegetable oil by homogeneous base catalyst. Table 1 summarizes the experimental matrix of preliminary experiments designed to initiate the iterative methodology according to 3 operating conditions. The sequence of iterative experiments will be presented and discussed with respect to parameter identifiability, interest of proposed new experiments, model structure and convergence of the performance criterion.

**Table 1.** Experimental matrix for preliminary experiments for ethanolysis of vegetable oil by homogeneous base catalyst

| Run | Reactor | Operating conditions | | |
|---|---|---|---|---|
| | | Reaction Temperature (°C) | NaOH amount (%) | Ethanol to oil molar ratio |
| 1 | Batch | 60 | 0.5% | 6 |
| 2 | Batch | 45 | 0.5% | 6 |
| 3 | Batch | 60 | 1% | 6 |
| 4 | Batch | 60 | 0.5% | 12 |
| 5 | Microreactor | 60 | 0.5% | 6 |
| 6 | Microreactor | 45 | 0.5% | 6 |
| 7 | Microreactor | 60 | 1% | 6 |
| 8 | Microreactor | 60 | 0.5% | 12 |

## 4. Conclusions

The results of the experimental strategy applied to ethanolysis of sunflower oil by homogeneous base catalyst and the second version of PIS confirm the importance of optimal experimental planning and the efficiency of a support software, but also highlight the role of the expert user for choice of model structure and evolutions.

## References

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***Keywords***
Process intensification; Computer-aided software; Ethanolysis of sunflower oil by homogeneous base catalyst