

Flow chlorination with SOCl_2

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CHEMICAL DEVELOPMENT & PRODUCTION

HEIA-FR

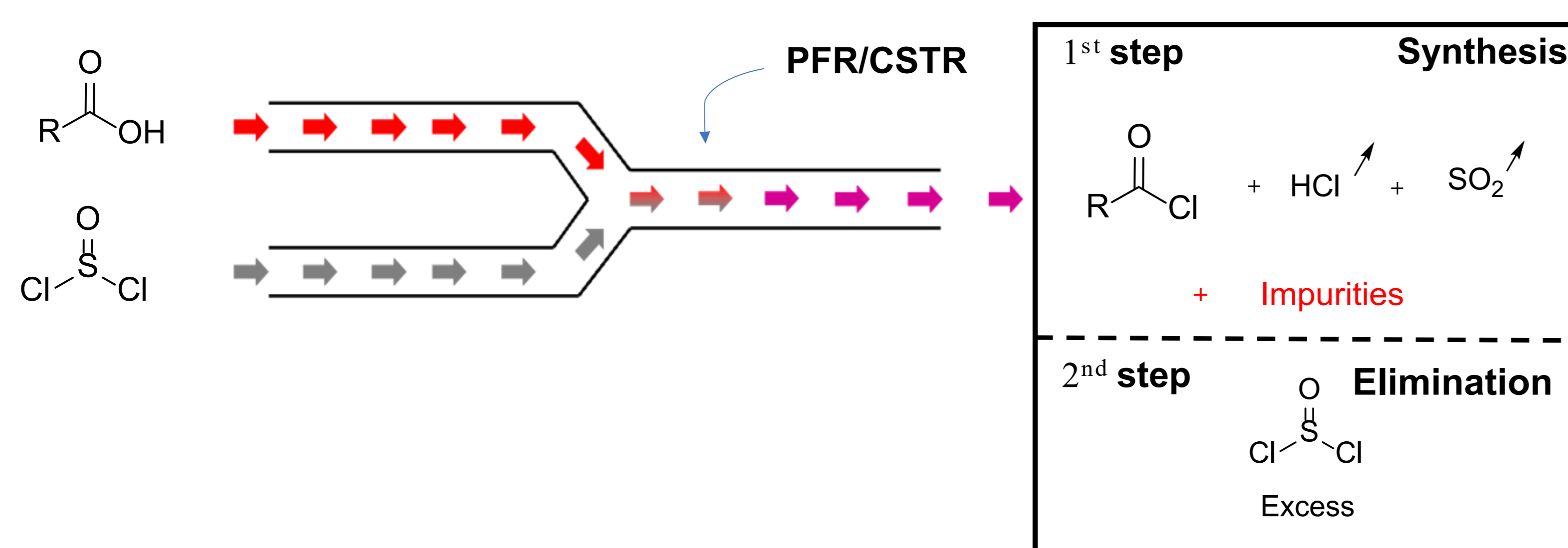
Advisor: Dr. Allemann Christophe // In collaboration with **Syngenta Monthey**

DESCRIPTION

This master's project is a collaboration between HEIA-FR and Syngenta Monthey and focuses on the development of a process flow of an intermediate reaction. The aim is to carry out in continuous mode a chlorination with thionyl chloride (SOCl_2), a highly reactive chlorinating agent. [1]

Initially, the project focus on the synthesis of a derivative of acyl chloride from a carboxylic. Various technologies are being tested, including the Plug Flow Reactor (PFR) and the Continuous Stirred-Tank Reactor (CSTR).

In the second part, the continuous elimination of residual SOCl_2 is studied. This can be achieved by falling film distillation directly linked to the reaction medium outlet or by reactive distillation as a process intensification.



[1] Phosphorus Sulfur Silicon Relat. Elem. 2021, 196 (8), 685-689.

OBJECTIVES

The main objective of this master's thesis is to assess the feasibility and competitiveness of the continuous process compared with the industrial batch process. Costs, productivity, yield, robustness and reproducibility must be considered when selecting the best set-up. To achieve this, a Design of Experiment (DoE) is carried out on the different technologies tested.

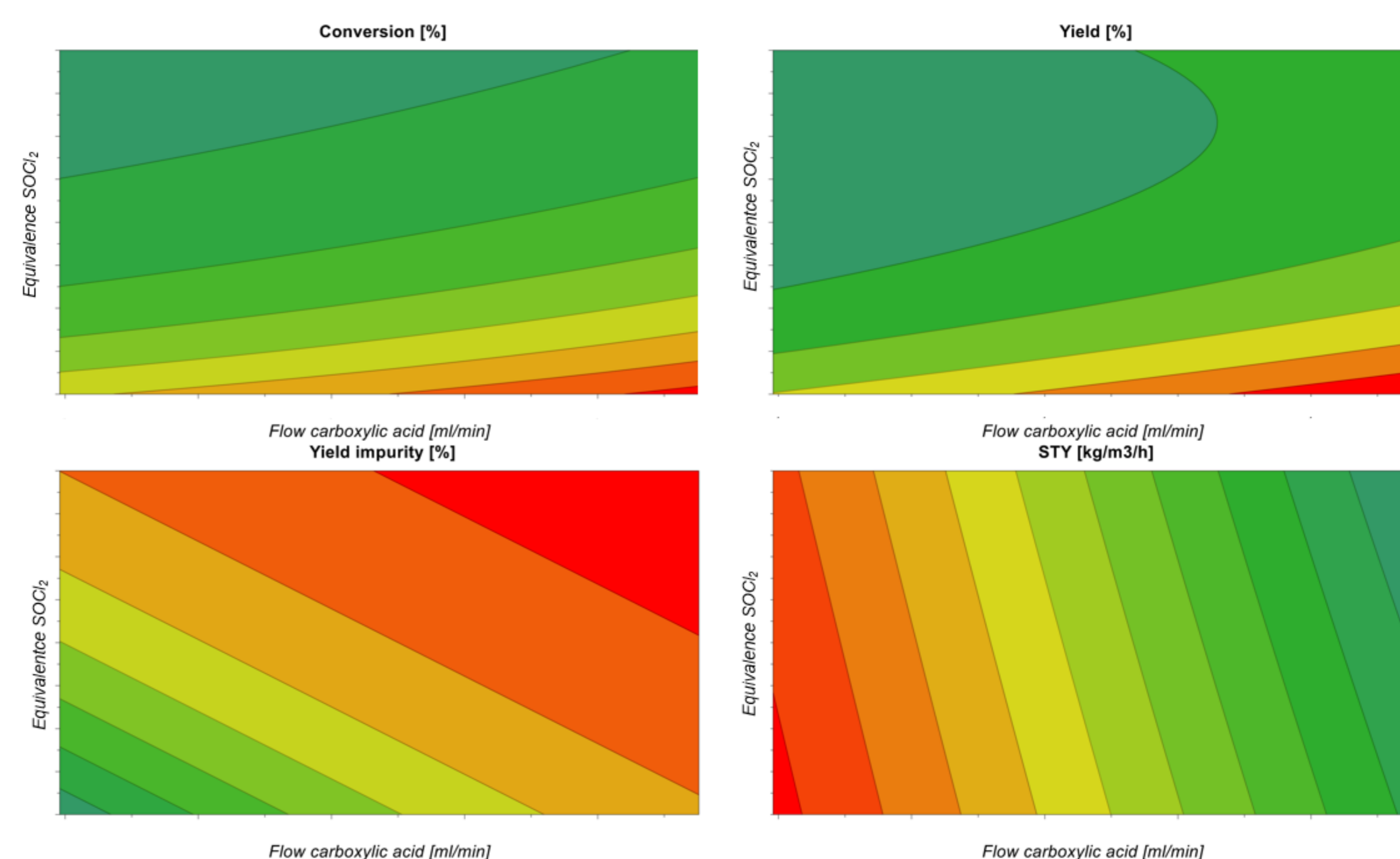
Then, an optimization is carried out to find the best reaction conditions. The second objective is to find an efficient downstream treatment method for continuously removing excess SOCl_2 from the reaction medium.

- ① Feasibility
- ② Competitiveness
- ③ Downstream processing

RESULTS

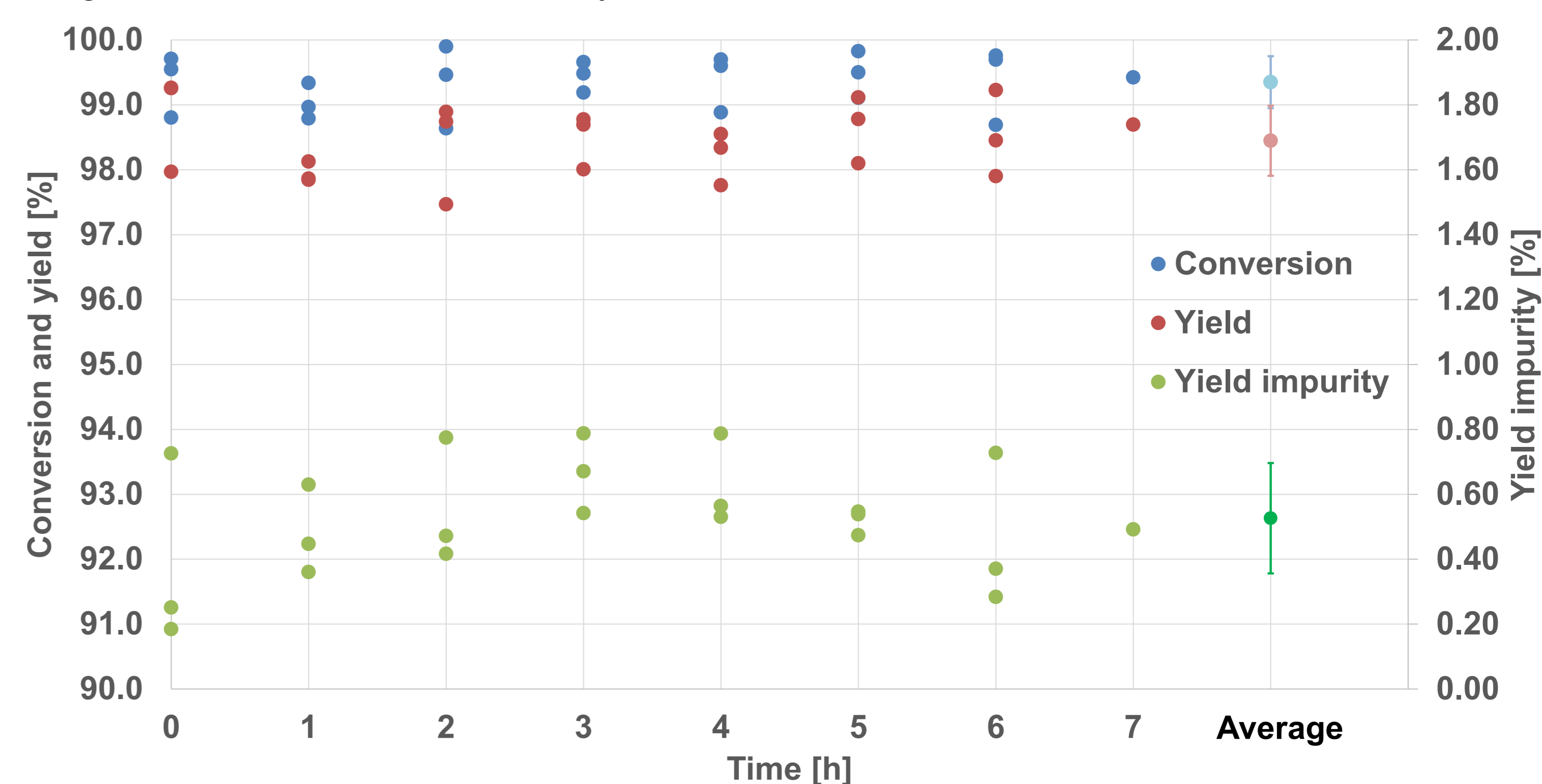
The MODDE 13 program has been used for the DoE and to visualize the impact of each variable on the desired response. [2] Optimization work can then proceed, finding the right compromise between cost, yield, productivity and impurity minimization.

[2] MODDE® - Design of Experiments Software. Sartorius



Example of graphs obtained during simulation

Once the best test has been obtained, the reproducibility and robustness of the installation is tested by carrying out several long-term tests (6 h or more) over several days. The best system found gave excellent results, with very little fluctuation.



Grouping of tests carried out over several days

Although the installations tested were created by hand for the purification. Residual SOCl_2 was reduced by a factor of 20 using a distiller simulating a falling film. In addition, reactive distillation is an intensive process with interesting potential, since complete conversions have been achieved.

CONCLUSION

The results obtained in continuous mode showed significant advantages over industrial batch mode. The savings in time, productivity and risk reduction are considerable. The next step would be to test the technology in continuous mode on a pilot scale, in order to confirm the results obtained in the laboratory.

Reactive distillation could also be implemented, offering the advantage of synthesizing the molecule of interest and eliminating residual SOCl_2 . However, it is important to note that recovery of excess SOCl_2 would be preferable to make the process greener.