PROSIMPLUS APPLICATION EXAMPLE

USE OF BATCHREACTOR AND BATCHCOLUMN IN PROSIMPLUS

THYMOL PRODUCTION

EXAMPLE PURPOSE
This example illustrates the use BatchReactor and BatchColumn software in ProSimPlus environment.

ACCESS	☑ Free Internet	☐ Restricted to ProSim clients	☐ Restricted	☐ Confidential

CORRESPONDING PROSIMPLUS FILES	*PSPS_EX_ENU-Thymol-Production.pmp3*
	Thymol Reactor.pbpr
	Thymol Distillation Column.pbpc

Reader is reminded that this use case is only an example and should not be used for other purposes. Although this example is based on actual case it may not be considered as typical nor are the data used always the most accurate available. ProSim shall have no responsibility or liability for damages arising out of or related to the use of the results of calculations based on this example.

www.prosim.net

TABLE OF CONTENTS

1. PROCESS MODELLING

1.1. Process description

Thymol is a phenol contained in the thyme oil and in the volatile essential oils of other plants. It takes the form of colorless crystals with a specific aromatic smell. It is soluble in alcohols, in fat and oil, but slightly soluble in water. It is notably used for its antiseptic action, its antibacterial properties and its antifungal effect, as well as to stabilize the pharmaceutical preparations.

This example deals with the thymol production. Thymol synthesis and distillation are operated in batch modes. The synthesis operating mode involves two steps. During the first step, one of the reactants is fed, and, during the second step, the reaction is continued with no feed. The thymol purification and the recovery of unreacted m-cresol are made with a batch distillation. The operating mode consists of five steps: filling of the column, infinite reflux, m-cresol cut, inter-cup and thymol cut. The technological elements in relation with the heating devices of the boiler are taken into account (external jacket and helical coil).

Detailed information on batch reactor and distillation simulations are available in a series of three examples dealing with the synthesis and the purification of thymol. The first example: « SIMKIN_EX_EN - Thymol » allows to identify the chemical reactions parameters. The second example "BATCHREA_EX_EN – Thymol" deals with the synthesis of thymol. The third example: « BATCHCOL_EX_EN - Thymol » deals with the thymol purification after its synthesis.

In this example, the simulation is done in the ProSimPlus environment, batch reactor and batch column simulations are made using respectively BatchReactor and BatchColumn run in the steady state simulation software ProSimPlus. The interest of this example is to show how it is possible to chain the simulation of a process integrating continuous parts and batch parts using ProSim software.

1.2. Process flowsheet
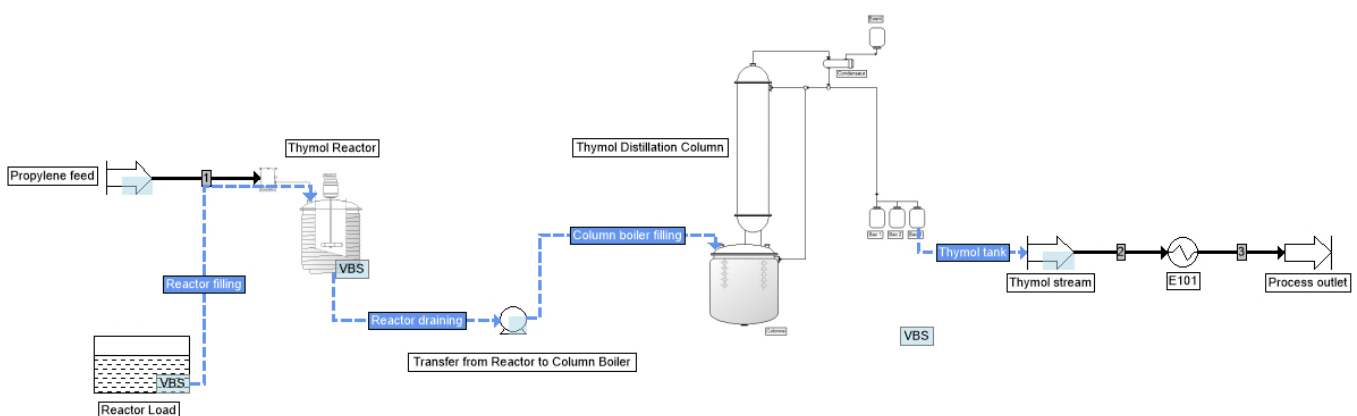
1.3.   Components

The components taken into account in the simulation are:

Name	Formula	CAS Number
Propylene[(*)]	C_3H_6	115-07-1
m-cresol[(*)]	C_7H_8O	108-39-4
Thymol[(*)]	$C_{10}H_{14}O$	89-83-8
3-methyl-2-isopropylphenol (3M2P)	$C_{10}H_{14}O$	-
1-methyl-3-hydroxy-5-isopropyl benzene (3M5P)[(*)]	$C_{10}H_{14}O$	3228-03-3
1-methyl-3-hydroxy-6-isopropyl benzene (3M4P)[(*)]	$C_{10}H_{14}O$	3228-02-2

Components with an asterisk are taken from the standard database of Simulis Thermodynamics. The thermophysical properties stored in this database are the DIPPR recommended values [ROW17].

The 3M2P component (3-methyl-2-isopropylphenol) has been created by cloning the thymol component from the standard database. Only the name, the CAS number, the normal boiling point and the vapor pressure have been modified:

- ✓ IUPAC name: 3M2P
- ✓ Specific name: 3-methyl-2-isopropylphenol
- ✓ CAS Number: 55000-01-6 (arbitrary number)
- ✓ Normal boiling point: 501,1 K
- ✓ Vapor pressure:
  - ○ Correlation: Equation #99
  - ○ T_{min}:   50 K
  - ○ T_{max}:   700 K

$$Ln(P^0) = 20,88 - \frac{7569}{T + 30,15}$$

In order to display the components acronyms in the interface instead of their full name in the standard database, the 3M2P, 3M4P and 3M5P acronyms are specified as "IUPAC name" of the corresponding components. This way, the names defined can be kept in **Specific name**.

1.4.   Thermodynamic model

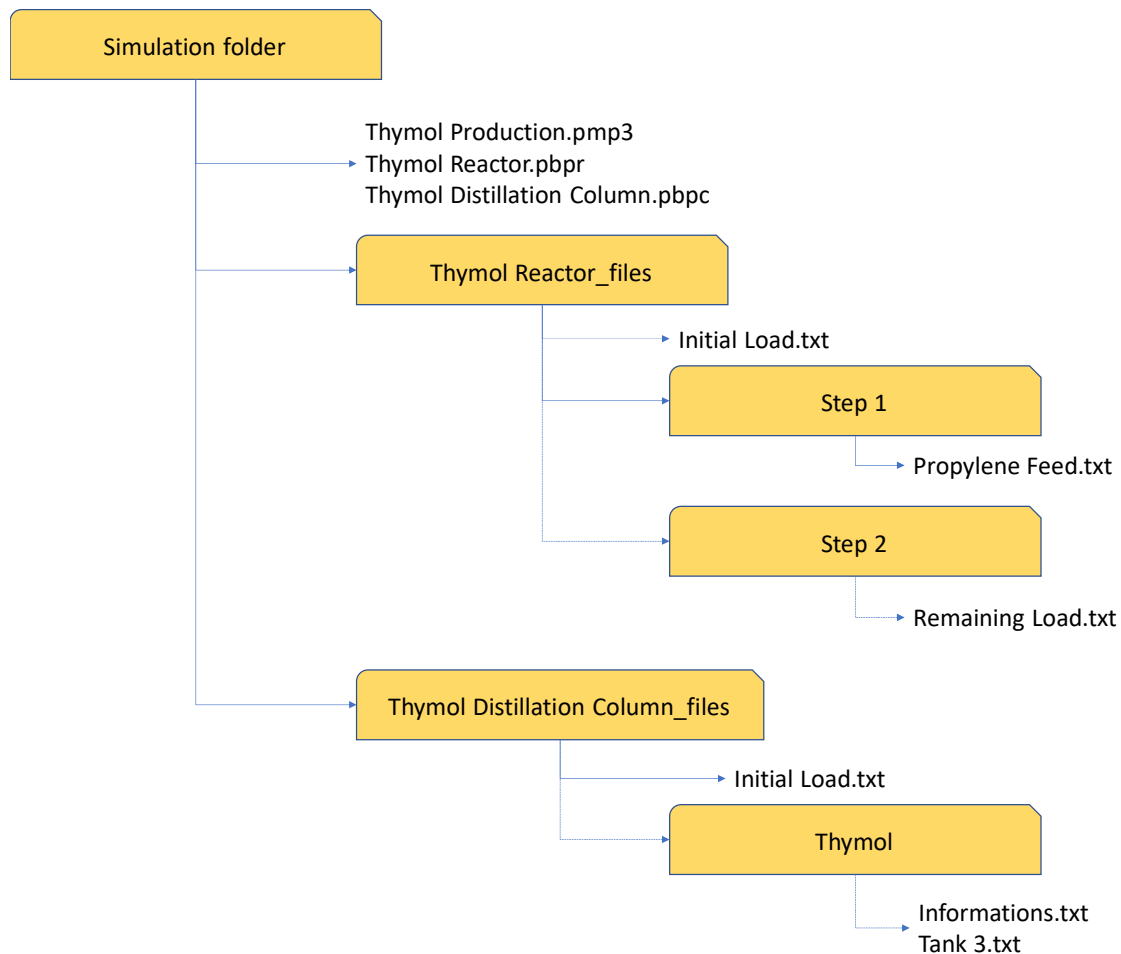In this example, the **Ideal** thermodynamic profile has been chosen.

Simulis Thermodynamics « calculators » described in ProSimPlus, BatchReactor et BatchColumn are the same in terms of components and thermodynamic profile

2. SIMULATION

2.1.   Process description

The process includes batch simulation modules (the "Thymol Reactor" reactor, and the "Thymol Distillation Column" column) as well as steady-state modules (the "Thymol stream" feed and the cooler "E101"). ProSimPlus, BatchReactor, and BatchColumn simulation files must be placed in the same working directory. For each batch unit operation, a directory with the same name as the unit operation simulation file suffixed with "_files" must be present. These folders contain the files that will be used to exchange information between ProSimPlus and the BatchReactor and BatchColumn software. The structure of the information is presented in the diagram below.

```
Simulation folder
│
├──→ Thymol Production.pmp3
│    Thymol Reactor.pbpr
│    Thymol Distillation Column.pbpc
│
├──→ Thymol Reactor_files
│         ├──→ Initial Load.txt
│         ├──→ Step 1
│         │         └──→ Propylene Feed.txt
│         └──→ Step 2
│                   └──→ Remaining Load.txt
│
└──→ Thymol Distillation Column_files
          ├──→ Initial Load.txt
          └──→ Thymol
                    └──→ Informations.txt
                         Tank 3.txt
```

The characteristics of the reactor load are defined in the Windows Script module of ProSimPlus "Reactor Load". The user specifies the load in the parameters of this module, which are then transferred to the "Initial Load.txt" file used by BatchReactor.

The ProSimPlus Feed Module "Propylene Feed" automatically changes propylene feed parameters during the "Step 1" reactor simulation step. The transfer of information between the data entered in this feed module and the "Propylene Feed.txt" file is done in the "Script" tab of the "Propylene Feed" module.

At the end of the Thymol synthesis, the reactor load is sent to the boiler of the column. This drain is represented by a ProSimPlus Windows Script module "Transfer from Reactor to Column Boiler" which retrieves the information generated by BatchReactor ("Remaining Load.txt" file) and transfers it to the "Initial Load.txt" file used by BatchColumn.

After the batch column simulation, the content of the "Thymol" distillation tank is used to generate a continuous feed. This information transfer is done in the "Script" tab of the "Thymol Stream" feed. It is assumed that the batches are chained so that this feed can be considered continuous.

The name of the BatchReactor or BatchColumn unit operation must be identical to the base name (name without extension) of the corresponding simulation file (« Thymol Reactor » for the file « Thymol Reactor.pbpr » and « Thymol Distillation Column » for the file « Thymol Distillation Column.pbpc »).

     www.prosim.net

2.1.1. Reactor

The reactor used for the thymol synthesis is a monophasic liquid reactor.
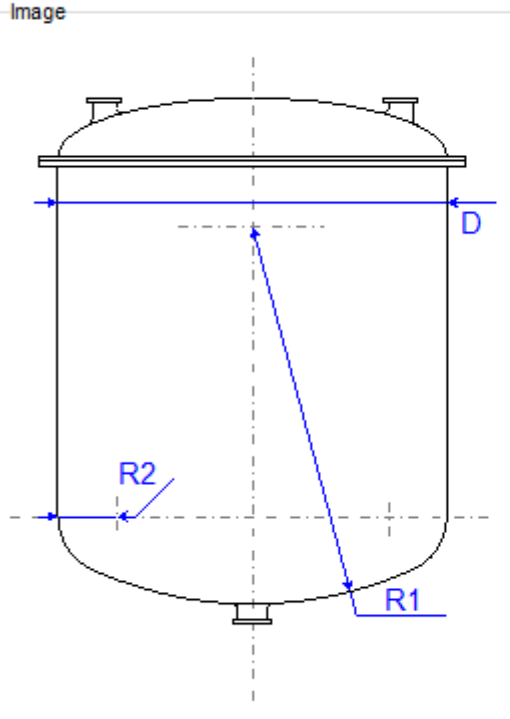
The initial conditions are presented in the following table:

Initial conditions	
Temperature	25°C
Pressure	12 atm
Initial load	
Total load	1 486 kg
Propylene	2% mass
m-cresol	98% mass

The vessel bottom geometry is described hereafter:
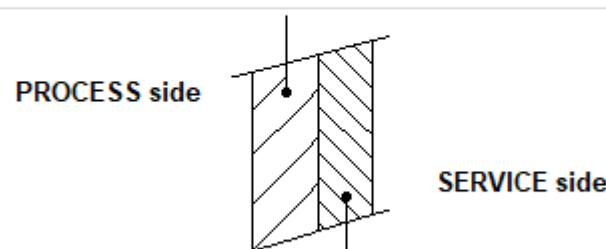
The reactor is made up with glass-lined steel.
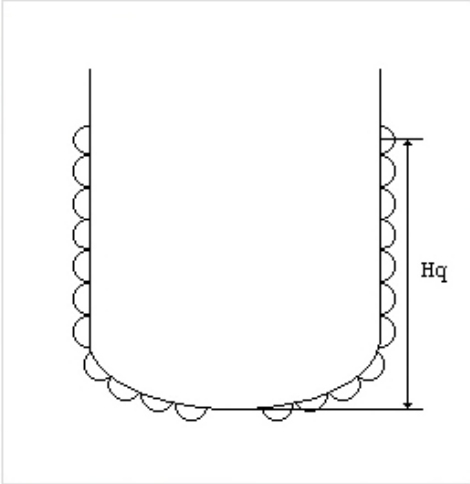
The steel thermal conductivity (material #2) is considered to be equal to 52,25 $W.m^{-1}.K^{-1}$ and the enamel thermal conductivity (material #1) to 1,161 $W.m^{-1}.K^{-1}$. The thermal conductivities are specified for each operating step.

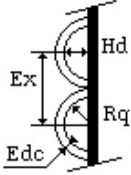The alarms are as follows:

	Volume	Temperature
Minimum	1 l	0°C
Maximum	3 m^3	40°C

2.1.1.1. Cooling device

The reactor is equipped with a wall heat exchanger (partial-pipe jacket), with the following characteristics:

The thermal fluid used is the same for the two operating steps:
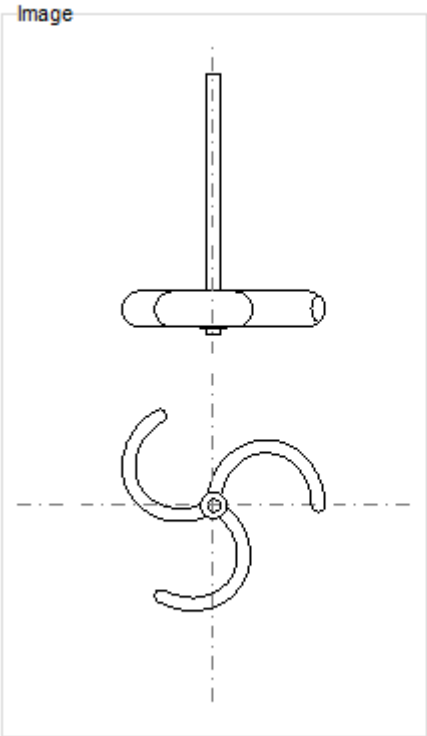
Heat transfer fluid (the same for the two steps)	
Type	Water
Mass flowrate (initial value)	1 500 kg/h
Inlet temperature	14°C

The water mass flowrate will be automatically adjusted in order to keep constant the reactor temperature as it has been defined in each operating step.

2.1.1.2.   Mixing device

The characteristics of the mixing device appear hereafter:

> ℹ️ The agitator height applies to the distance between the agitator and the vessel bottom.

The rotation speed of the agitator (60 rpm) is defined in each operating step (same speed for the two steps).

2.1.1.3.   Feeds

A continuous flow of propylene (reactant) is supplied during the first step:

Temperature	25°C
Pressure	12 atm
Propylene flowrate	39 kg/h

2.1.1.4. Operating mode

The operating mode is made up with two steps. During the first step, the second reactant (propylene) is added. The reaction starts. During the second step, the reaction is carried out but without propylene feed. These two steps are carried out at constant temperature, by acting on the utility fluid flowrate. The operating parameters are given in the following table:

Parameter	First step	Second step
Type of step	Specified TR with thermal device	
Reactor temperature	25°C	
Reactor pressure	12 atm	
Propylene feed	Open	Closed
Stop event	Time elapsed since the beginning of the step: 10 h	Time elapsed since the beginning of the step: 30 h

The scenario is presented on the left of the following screenshot and the flowsheet on the right part.
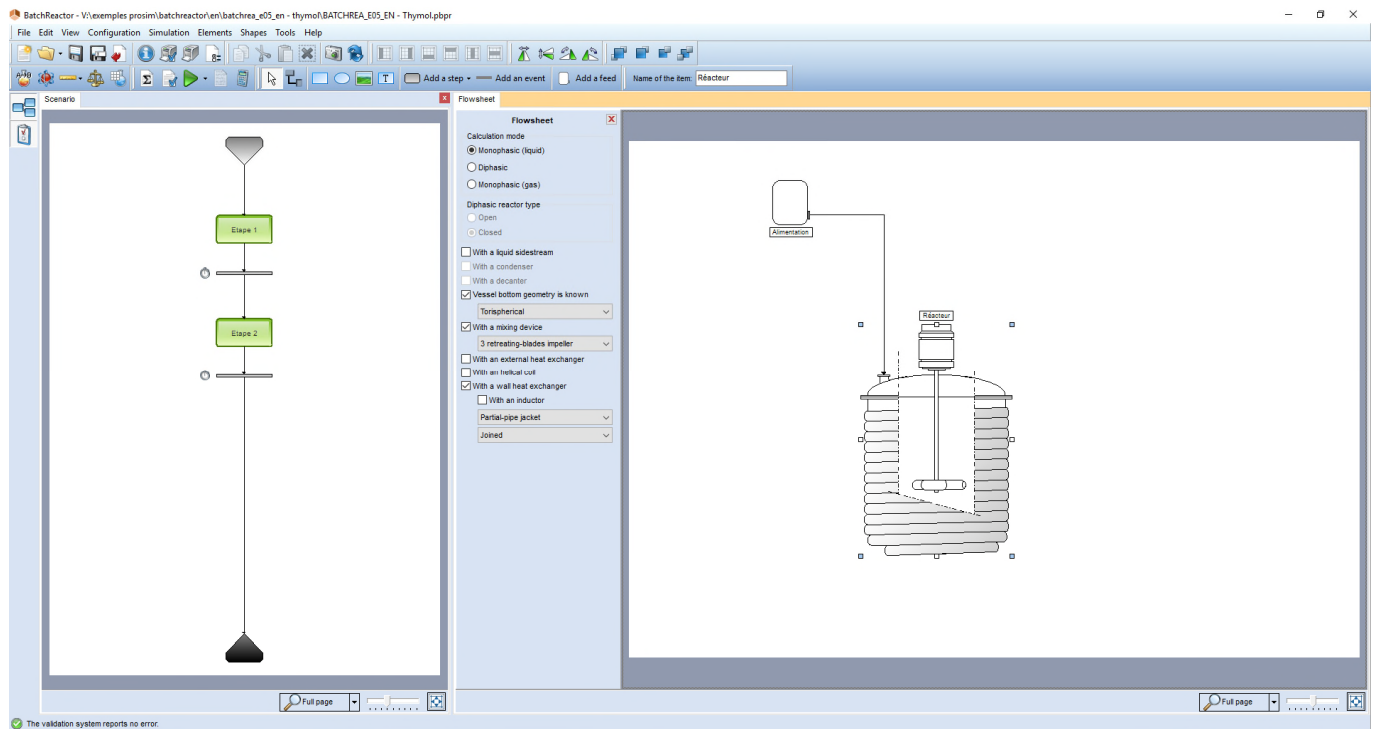
2.1.2. Distillation column

The column is made up with 56 theoretical stages, including the boiler and the condenser.

Three collection tanks will be used during the distillation step.

The initial load is detailed below. It corresponds to the content of the reactor at the end of the synthesis without the propylene (see the "BATCHREA_EX_EN – Thymol" example).

- ✓ Temperature   25°C
- ✓ Total mass   1 825 kg
- ✓ Composition   m-cresol   27,9541% wt.
                    Thymol   24,3378% wt.
                    3M2P   18,7774% wt.
                    3M5P   2,3200% wt.
                    3M4P   26,6107% wt.

The liquid hold-ups are supposed to be constant all along the distillation:

- ✓ Condenser   5 l
- ✓ Column   4 l for each theoretical stage

The pressure profile is also supposed to be constant all along the distillation process
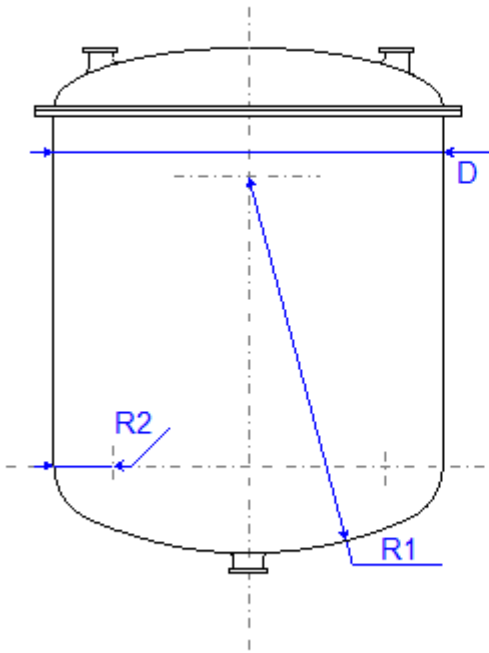
- ✓ Condenser   40 mmHg
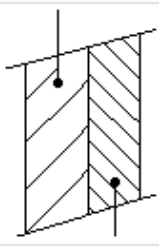- ✓ Pressure drop   10 mmHg

2.1.2.1.   Condenser

The condenser is supposed to be ideal total all along the distillation process.

2.1.2.2.   Boiler

The column's boiler characteristics are detailed below.

Image	Type of vessel bottom geometry
	Torispherical

Parameters

Parameter	Value
Number of baffles	0
Vessel diameter (D)	1.2 m
Vessel bottom height (H)	0 m
Curve radius #1 (R1)	1.2 m
Curve radius #2 (R2)	0.12 m
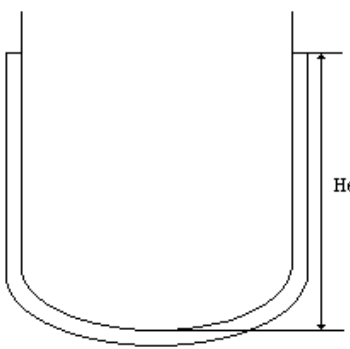
With a wall material defined as follows:

Number of wall materials	1

Material #1

Thickness	10 mm
Weight	0 kg

PROCESS side   SERVICE side

Material #2

Thickness	0 m
Weight	0 kg

The thermal conductivity of the wall is considered to be equal to 19 $W.m^{-1}.K^{-1}$. The thermal conductivity is specified for each step.

2.1.2.3.　Boiler heating device

The column's boiler is equipped with a wall heat exchanger (external jacket) with the following characteristics:
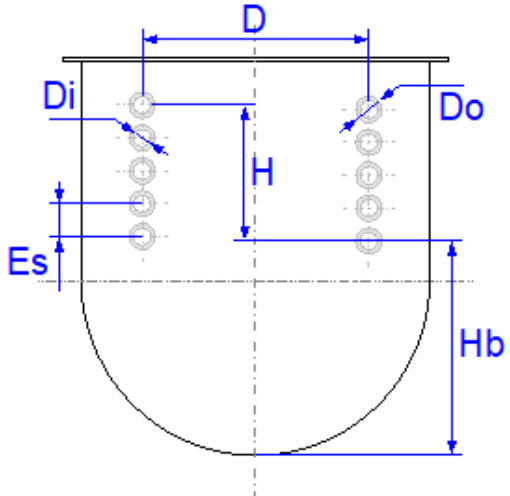
And with an immersed heat exchanger (helical coil) detailed below:

The thermal fluid used is the same for both systems and is described in the table below:

Heat transfer fluid (the same for all the steps)		
Exchanger	Wall heat exchanger	Helical coil
Fluid	Gilotherm DO RP (150 300)	
Flowrate	400 kg/h	1 350 kg/h
Inlet temperature	300°C	

2.1.2.4. Operating mode

The operating mode is made up with five steps. During the first step, the column is filled. A second step at infinite reflux allows to classify the products in the column. The third step allows to recover the maximum quantity of m-cresol to recycle it. The next step, an intercut, allows to eliminate mainly the 3M2P. The last step is the thymol production. The operating parameters of these steps appear in the table below.

| Parameter	Step 1 Filling	Step 2 Infinite reflux	Step 3 m-cresol cut	Step 4 3M2P elimination	Step 5 Thymol cut
Type	Filling	Infinite reflux	Distillation		
Operation	Variable heat duty				
Reflux ratio	-		2,5	50	
Collection tank	-		Tank #1	Tank #2	Tank #3
Stop event	Column filled	Step duration = 40 min	m-cresol content in the tank #1 < 99,9% wt.	Duration of the step = 26 h	Thymol content in the tank #3 < 87% wt.

The scenario is presented on the left of the following screenshot and the flowsheet on the right part.
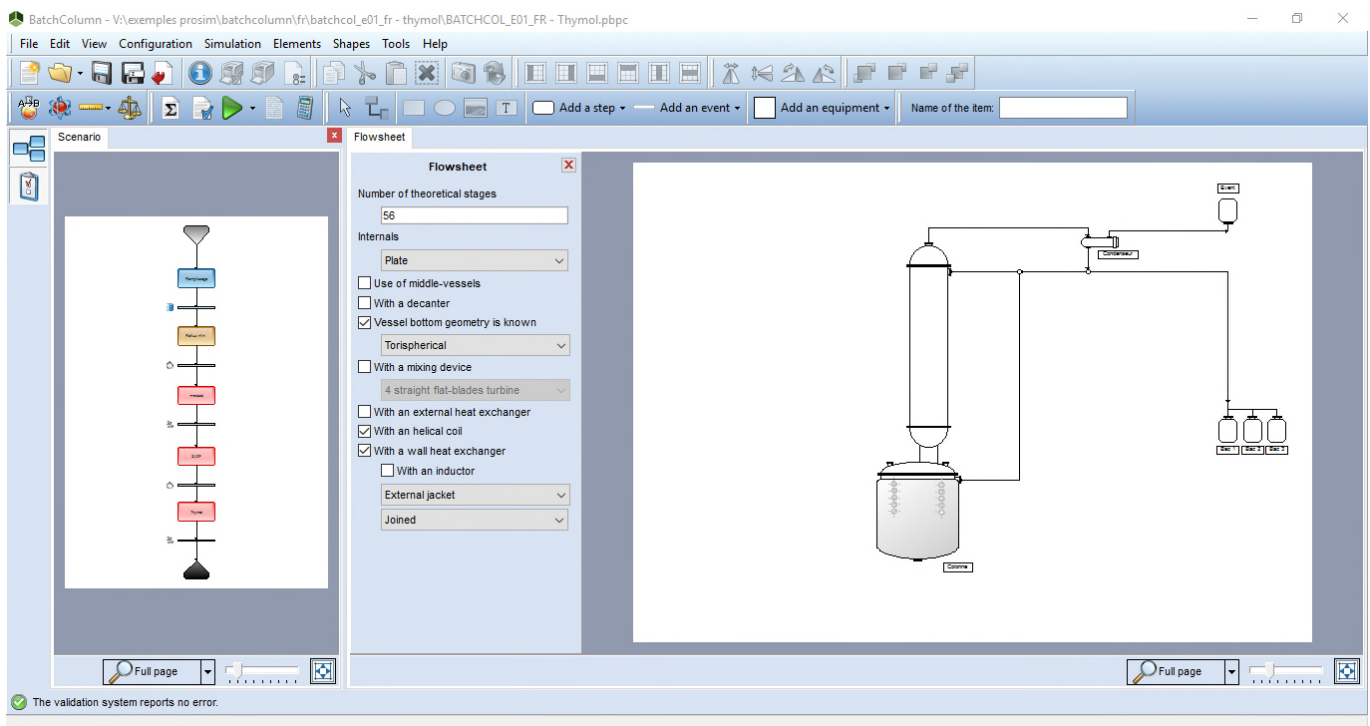
2.2. Results

2.2.1. Access to batch simulation results

The user can simply access the BatchReactor and BatchColumn simulation file by right-clicking on the desired unit operation and using the associated scriptlet.
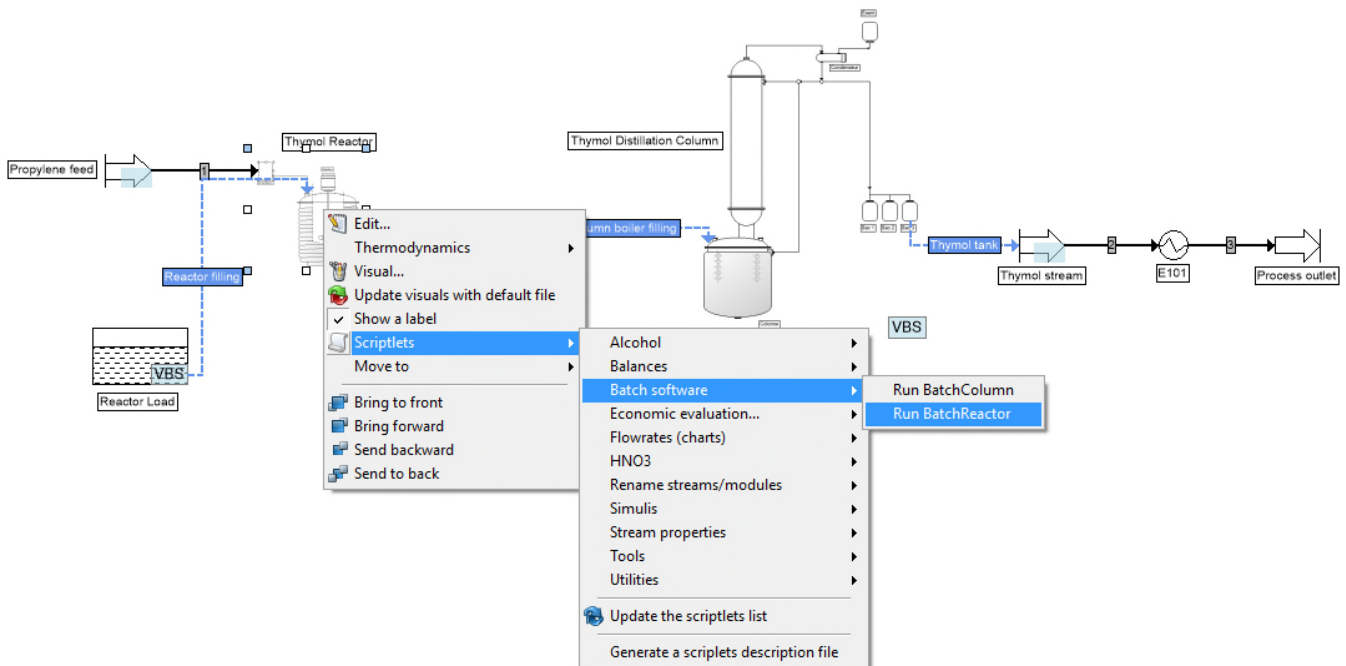

In order for the scriptlet to directly open the BatchReactor or BatchColumn file, the name of the unit operation must be identical to the name of the corresponding simulation file.

2.2.2. Reactor

The mass fraction of m-cresol decreases throughout the operation, as this reactant is present only in the initial load. The mass fraction of propylene increases during the first step (10h): as this reactant is fed with a flowrate that exceeds the quantity consumed, it then accumulates during this period. After the feed stops, it is consumed and disappears. The mass fracions of the reaction products (thymol, 3M2P, 3M5P, 3M4P) increase all along these two steps.

Mass fractions in the reactor
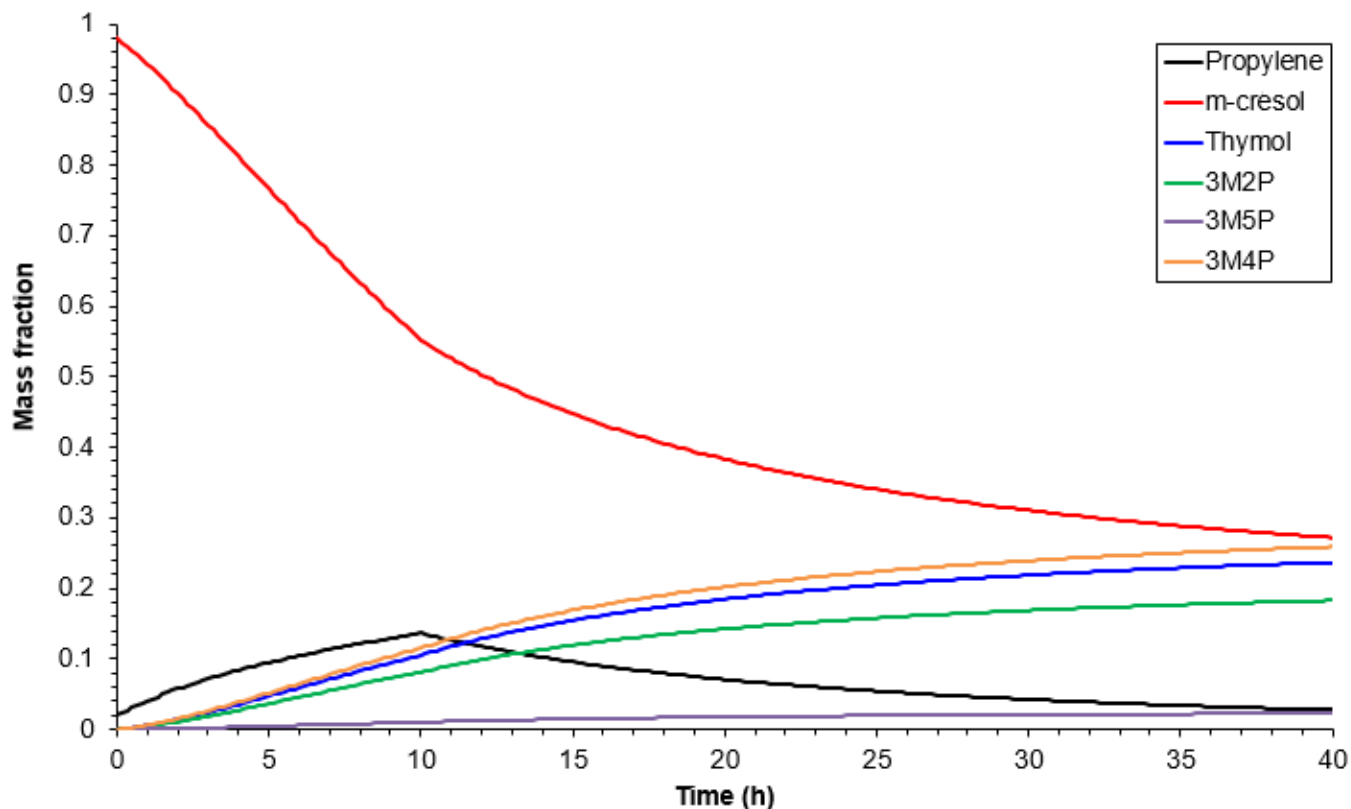
The following graph shows the evolution of the water flowrate necessary to keep the reactor isothermal at 25°C. For 7 hours, the necessary flowrate of utility increases as the reaction starts strongly due to the propylene addition. Then the flowrate decreases, as the m-cresol is consumed decreases, and the reaction slows down. At the beginning of the second step (i.e. after 10 hours), this change is faster as there is no longer propylene supply.

Utiliy flowrate (water) necessary to keep the reactor isothermal at 25°C
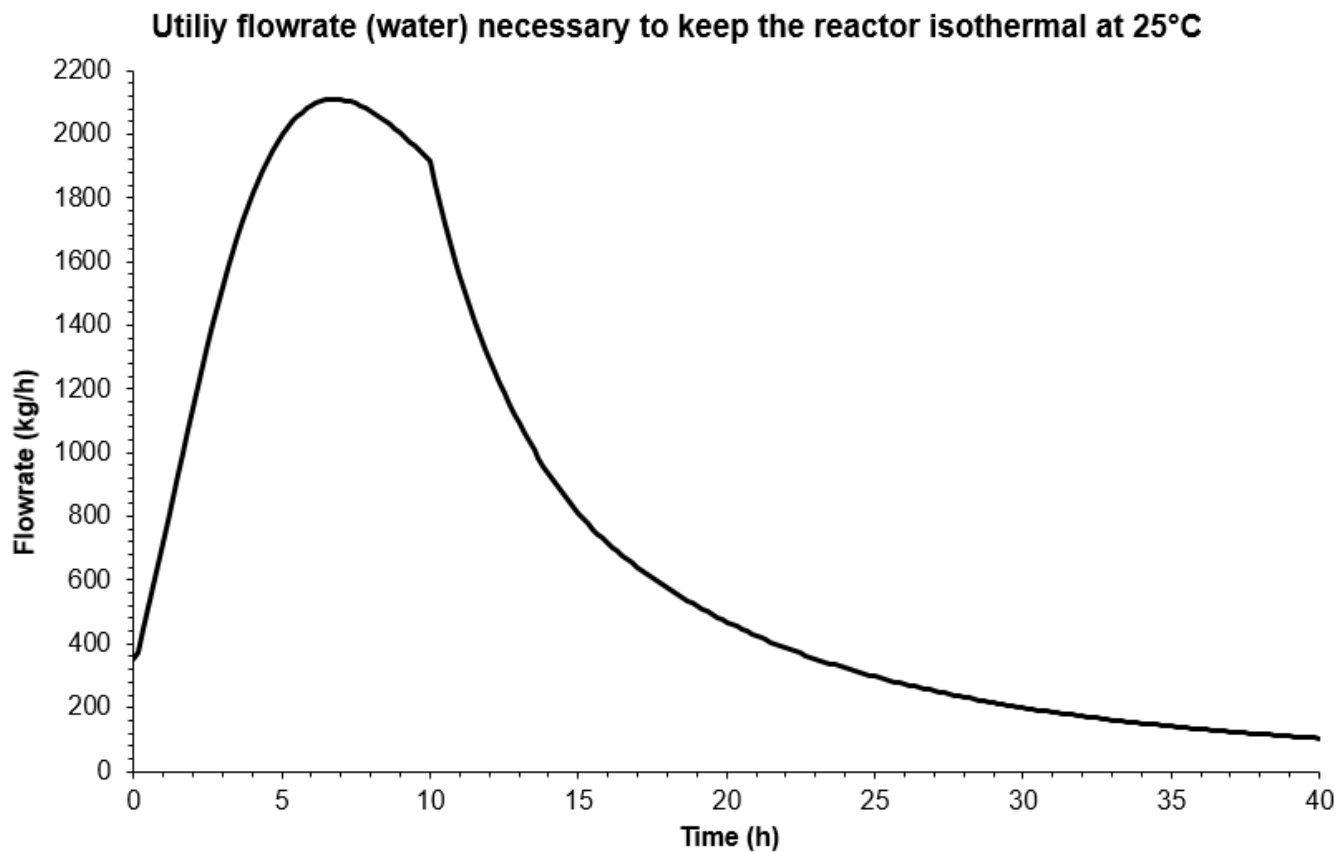
2.2.3. Distillation column

During the filling step, there is no liquid distillate. During the third step, the liquid distillate consists mainly of m-cresol. The m-cresol cut is stopped as soon as the content in 3M2P begins to be significant. The 3M2P cut contains 3M2P, the remaining m-cresol and the thymol that begins to be at the top of the column. The thymol cut is stopped when the collection tank meets the specification requirement.

Liquid distillate compositions evolution
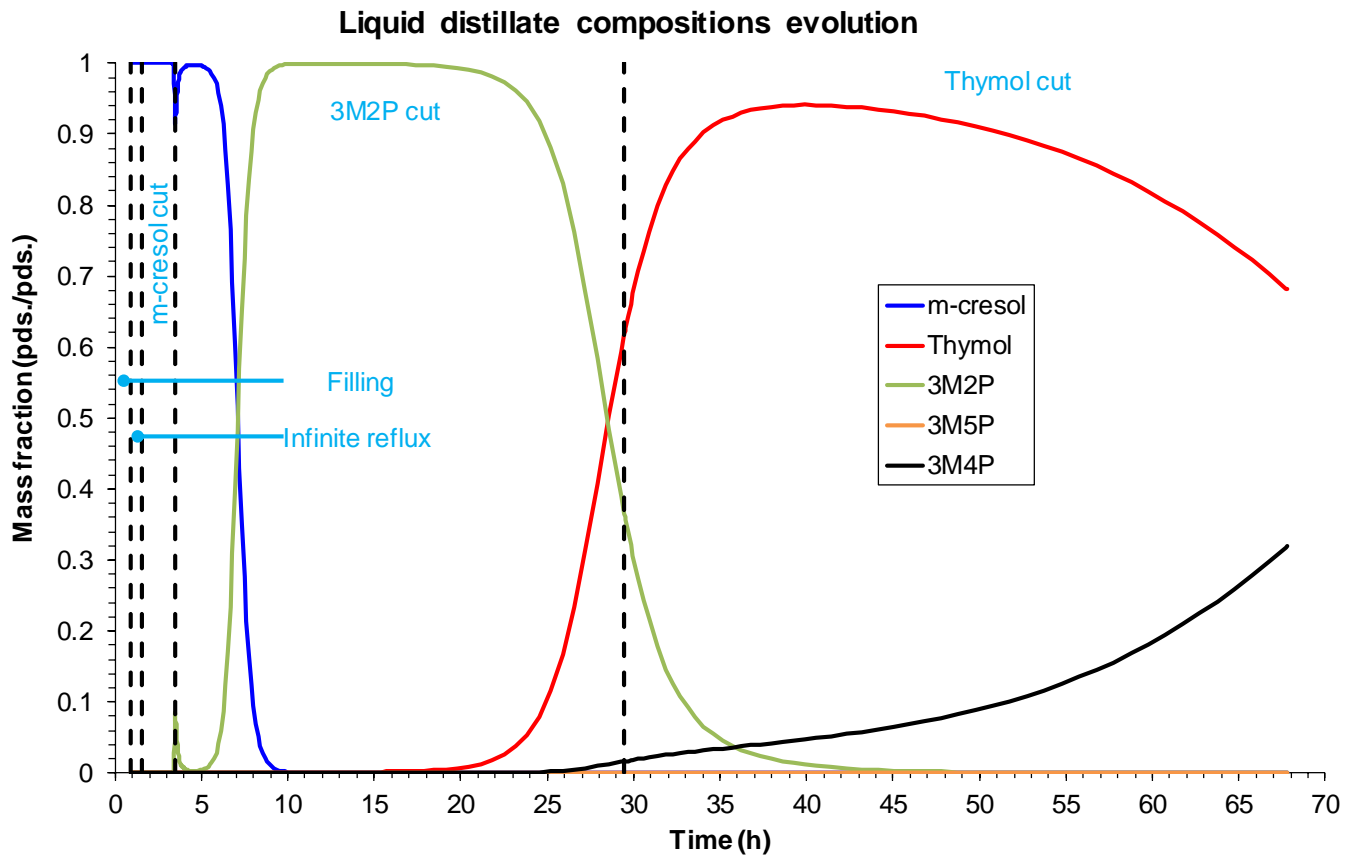
The table below shows the content of the collection tanks, of the boiler and of the liquid holdups (condenser and plates) after the operation is completed. The 3M5P and 3M4P mainly stay in the boiler, and the liquid holdups in the column.

	Tank 1	Tank 2	Tank 3	Boiler	Liquid holdups
Mass (kg)	405	408	401	376	184
Composition (wt. %)					
m-cresol	99,90	13,51	0	0	0
Thymol	8 ppm wt.	5,89	87,00	5,69	27,01
3M2P	0,10	80,47	3,43	0,1 ppm wt.	1 ppm wt.
3M5P	0	Traces	Traces	10,89	0,75
3M4P	0,4 ppm wt.	0,13	9,57	83,42	72,24

3. REFERENCES

[ROW17]   ROWLEY R.L., WILDING W.V., OSCARSON J.L., GILES N.F., "DIPPR® Data Compilation of Pure Chemical Properties", Design Institute for Physical Properties, AIChE, New York, NY (2017)

3. REFERENCES