

# Process Control of Isobutane-Butene Alkylation Unit

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*This paper presents the assessment of the dynamic behavior and the control system response of the isobutane-butene alkylation process, via dynamic simulations performed in Aspen Dynamics. The steps for developing the dynamic models, the control structures and the controllers tuning are discussed. The transient behavior of the plant when different disturbances are considered is evaluated. Two flowsheets are investigated – one with three separation columns and the other one with two separation columns for product purification. The results are presented in graphical form and a comparison between the two flowsheets is performed. Methods for varying the plant throughput and for controlling the product purity are provided.*

*Key words: alkylation, process control, dynamic simulation, reactor-separation-recycle*

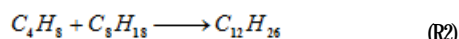
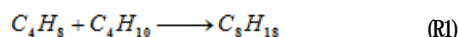
## Alkylation process

The concerns regarding the environment protection are higher and higher and the regulations on the conventional fuels are becoming year by year more severe. Therefore, the requirements for cleaner burning fuels have gradually increased.

The alkylate is a mixture of isoalkanes ( $C_8$  components), containing from 75 to over 150 different isoparaffins isomers [1], mainly 2,2,3-, 2,2,4-, 2,3,3- and 2,3,4- trimethyl pentane [2]. Isobutane alkylate has proved to be a suitable blending component for gasoline, which matches the current required properties: the vapor pressure limits and the contents of aromatics, olefins, nitrogen and sulfur components [3].

The alkylate is obtained by the reaction (R1) between isobutane and the butenes coming from the FCC processes. Several side reactions occur, out of which, the most significant is the reaction (R2) of alkylate with the butene, leading to heavier components ( $C_{12}$ ).

The reactions take place in a liquid phase stirred reactor (CSTR), in the presence of a strong acid catalyst: sulfuric or hydrofluoric acid. Due to the high level of toxicity associated with hydrofluoric acid, in the last decades, the sulfuric acid processes started to be preferred [3].



The main technologies for the production of alkylate, widely discussed in other publications [1, 3-6] involve a reaction step in a stirred reactor where cooling is achieved by heat transfer to a suitable refrigerant or by evaporation of a part of the reaction mixture, a liquid-liquid acid / hydrocarbon separation step, and a purification unit, usually consisting of several distillation columns for reactants recovery, removal of n-butane inert and product purification. The recovered reactants are recycled back to the reaction step.

Reference [4] presents the design and control of an alkylation plant, the heat of reaction being removed by auto-refrigeration through boiling of the organic phase and reactant / products / inert separation being achieved by a sequence of two distillation columns. In [6], where a flowsheet including a column with three product streams (with n-butane as liquid side-stream) had been described, the author claimed that it provided the least amount of

control over n-butane quality, being contaminated with isobutane and isopentane.

Dimian and Bildea [2] considered the design and control of an alkylation plant, using isobutane and butene of high purity as raw materials. The heat of reaction was removed using a suitable coolant, while the reactants / products / inert separation was achieved in three distillation columns. Dragnea and Bildea [3, 5] presented an optimized flowsheet, where the raw materials were hydrocarbon mixtures found in industry and the reaction took place in a series of three cooled stirred reactors. This paper continues the endeavor by a comprehensive study of the alkylation unit process control. Thus, a control structure is developed and the transient behavior of the plant is determined, emphasizing the controllability and flexibility of the distillation units. An alternative design of the purification unit, consisting of two distillation columns for product purification, is also presented and evaluated.

## Process control

An industrial plant should always be able to cope with frequent upsets such as sudden flow and / or composition changes, temperature / pressure variations, catalyst deactivation and many others. Stable operation, preferably at the desired operating point, should always be guaranteed. To achieve this goal, control systems are designed, ensuring plant safety, product specifications and profitability.

Process control is a multi-disciplinary field, where the process objectives, process constraints, instrumentation functionality and operational experience come together to maintain a safe, stable and optimum operation. The process control activities are related to the operational control and instrumented safeguarding. Operational control involves all manual and automated actions to properly operate the process and keep the process within its operating window. Instrumented safeguarding involves all unscheduled instrument actions which are designed to bring the process in a safe state if it moves out of its operating window and towards an unsafe situation. Instrumented safeguarding consists of the protection against personal injury, equipment and environmental damage and production loss.

This paper presents the development, the evaluation and the results of the operational control of the isobutane-butene alkylation plant. In this regard, the steady state Aspen Plus simulations of the alkylation unit are converted

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into dynamic flowsheets; control systems are developed and implemented in the dynamic flowsheet. The response of the plant / control system to different disturbances is assessed through dynamic simulations, performed in Aspen Dynamics.

### Dynamic simulation

Dynamic simulation of a chemical process greatly helps to understand the transient behavior. Aspen Dynamics, which is tightly integrated with Aspen Plus, is widely used for process design and control. It can automatically initialize dynamic simulation using the steady state results of Aspen Plus simulation [7, 8].

The performance / response of a control system depends on two main factors: the process characteristics – gain, time constant and dead time – which are determined by the design / size of the equipment (e.g. for a given flowrate, the smaller the volume, the faster the transient response, [8, 9]) and the controllers tuning parameters ( $K$ ,  $T_r$ ,  $T_d$ ). The inventories of material contained in all pieces of equipment affect the dynamic response [9]. Therefore, the size of all equipment must be determined and specified.

The dynamic simulation can be developed either as a pressure-driven or as a flow-driven model [7]. Pressure-driven simulation (fig.1) is more realistic as it includes all pumps and compressors needed to provide the pressure difference required for the flow of material. Valves must be installed where needed for control purposes and their characteristics specified.

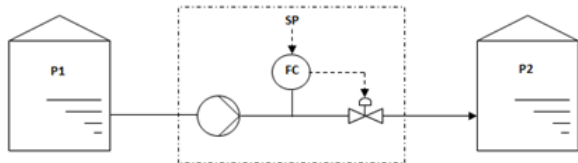


Fig. 1. Pressure-driven model

For the flow-driven simulations however (fig. 2), no such arrangements are required. The flowrate can be set to any desired value without any concern regarding how this is achieved. It works as the elements presented in the box of figure 1 would be designed and would work perfectly. The flow-driven simulation, which does not require sizing all pumps and control valves, has a much easier set-up than the pressure-driven simulation [7]. For the purpose of defining the main controllers and evaluating the transient behavior of the alkylation plant during a conceptual phase, the flow-driven simulation is a proper alternative.

The transient behavior of two flowsheets, presented in figure 3 and figure 6, is evaluated. The first flowsheet includes three separation columns for product purification - for light inert removal (propane), for the separation of unconverted reactants (isobutane and butene) from heavy

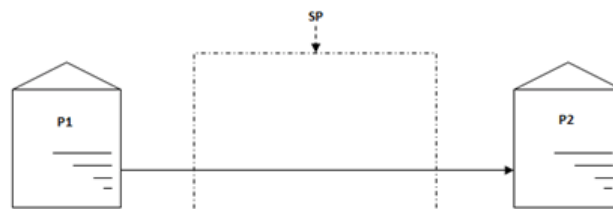


Fig. 2. Flow-driven concept

inert (n-butane) and products, and for the separation of n-butane from the product (isooctane and dodecane), respectively. The second flowsheet includes only two separation columns: one for separating the propane as vapor in the overhead stream, unconverted reactants as a side stream and n-butane plus product in the bottom stream, and one for the separation of n-butane from the product.

The sizes of the main equipment - reactors, separation columns, and heat exchangers - are also presented in fig. 3 and figure 6. The optimization procedure and sizing details are presented in reference [3]. In addition, the sizes of the reflux drums and bottom sumps were determined to allow for 5 min of liquid holdup when the vessel is 50% full, based on the total liquid leaving the vessel. Several controllers needed to keep the plant within the operating window are presented in the following section. They can be also seen in figure 3 for Flowsheet 1 and in figure 6 for Flowsheet 2.

### Control structure

#### Flowsheet 1

The control structure of Flowsheet 1 of the alkylation plant is presented in figure 3.

The material balance of the steady state flowsheet corresponding to the figure above is presented in table 1.

During the steady state evaluation of the alkylation process [5] it was determined that control of the reactor inlet flowrates was preferred to control of the plant inlet flowrates, avoiding snowball effects. Therefore, the fresh impure butene stream 2 containing some isobutane and inert components (propane and n-butane), is kept on flow control at the desired value; so is stream 1, the combined streams containing fresh and recycled isobutene.

Butene is the limiting reactant of the system, almost entirely consumed in the reaction. This allows having one recycle stream, rich in isobutane. Consequently, the fresh isobutane stream is used to control the level in the feed drum. When the recycle increases due to isobutane feed in excess of the stoichiometric amount, the fresh isobutane flow is decreased, and vice-versa.

The inventory in the reactors is controlled by manipulating the liquid outlet flowrate, while the temperature is maintained adjusting the reactor duty (flow rate of coolant through the reactor jacket).

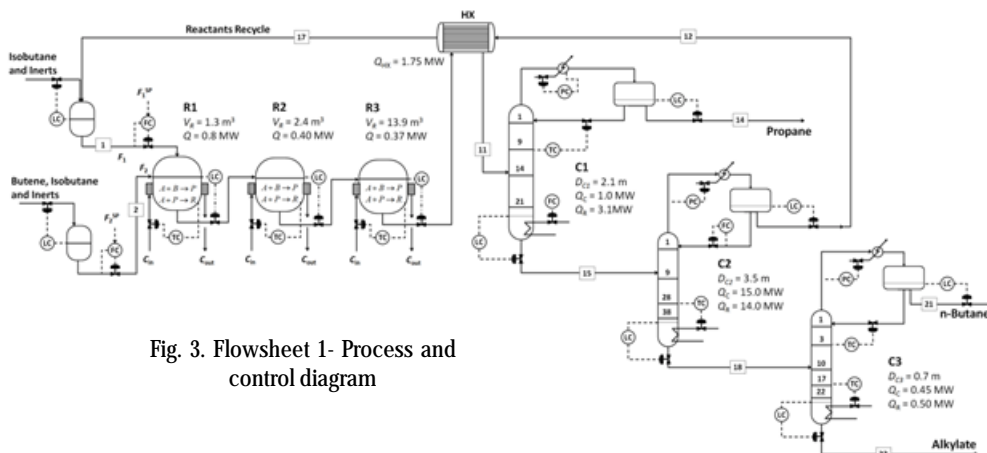


Fig. 3. Flowsheet 1- Process and control diagram

**Table 1**  
 FLOWSHEET 1 - STEADY STATE MATERIAL BALANCE

	Streams									
	1	2	11	12	14	15	17	18	21	22
<b>Mole Flow [kmol/h]</b>										
Butene	0.0	52.5	2.8	2.8	0.0	2.8	2.8	0.0	0.0	0.0
Isobutane	29.0	21.0	1594.6	1593.9	0.2	1594.4	1593.9	0.4	0.4	0.0
Isooctane	0.0	0.0	46.3	0.0	0.0	46.3	0.0	46.3	0.3	46.0
Dodecane	0.0	0.0	3.1	0.0	0.0	3.1	0.0	3.1	0.0	3.1
Propane	3.1	5.4	74.0	65.5	8.5	65.5	65.5	0.0	0.0	0.0
n-Butane	27.9	27.0	233.6	178.7	0.0	233.6	178.7	54.9	54.8	0.0
<b>Total Flow [kmol/h]</b>	60	106	1954	1841	9	1946	1841	105	56	49
<b>Total Flow [kg/h]</b>	3444	5974	115495	106077	386	115109	106077	9032	3246	5786
<b>Temp. [°C]</b>	15.0	15.0	38.1	44.3	23.4	63.5	20.4	89.7	24.8	138.4
<b>Press. [bar]</b>	6.0	6.0	11.0	6.0	9.0	9.2	6.0	6.3	2.4	2.6
<b>Vapor fraction</b>	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

For each column pressure and level loops are needed for inventory control; therefore, all three columns are provided with condenser pressure controller and level controllers for the reflux drums and column sump sections. The pressure is controlled in reverse action by the condenser duty ( $Q_c$ ), while the inventories are maintained in direct action by the product outlet flowrates (LV-configuration). This configuration for the level control is recommended for relatively small reflux ( $L/D$ ) and boil-up ( $V/B$ ) ratios, to avoid flow saturation [10].

The control particularities for each column are explained below.

The first column is designed to separate propane from the reactor effluent in the overhead stream. In order to prevent any heavy components carryover in the distillate flow, which could lead to loss of reactants and subsequently increased production costs, a temperature controller is

provided on tray 9, which manipulates the reflux flow in direct action. The reboiler duty for this column is fixed.

The location of all temperature-control trays is based on the *sensitivity criterion*, which consists in finding the tray where there is the largest change in temperature for a change in the manipulated variables (reboiler duty  $Q_r$  or reflux ratio  $RR$ ) [9]. The temperature profiles for different values of the manipulated variables are plotted and presented in figure 4, for all three columns.

The open loop steady state gain ( $K_{olSS}$ ), defined as the ratio between the temperature change ( $\Delta T$ ) and manipulated variable change (e.g. reboiler duty  $Q_r$ , reflux ratio  $RR$ , etc.) [9, 11] is plotted for each column. The zone where it reaches the highest value, for certain deviations of the manipulated variable is the proper location to install the temperature sensor.

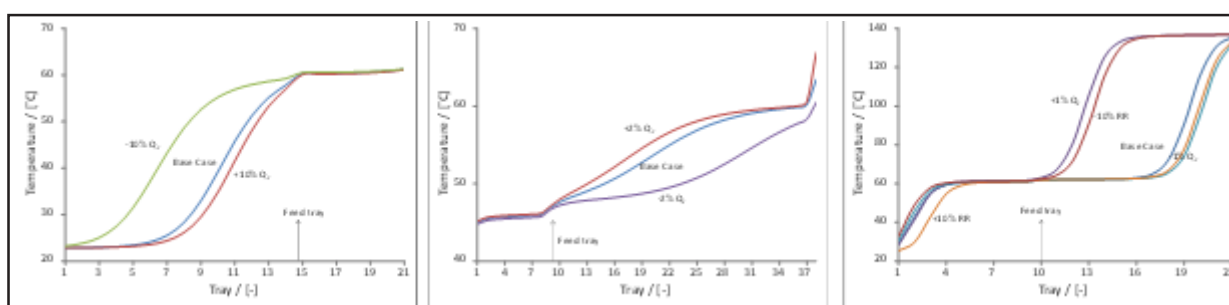


Fig. 4. Flowsheet 1 - base case and perturbed temperature profiles: column C1 (left), column C2 (middle), column C3 (right)

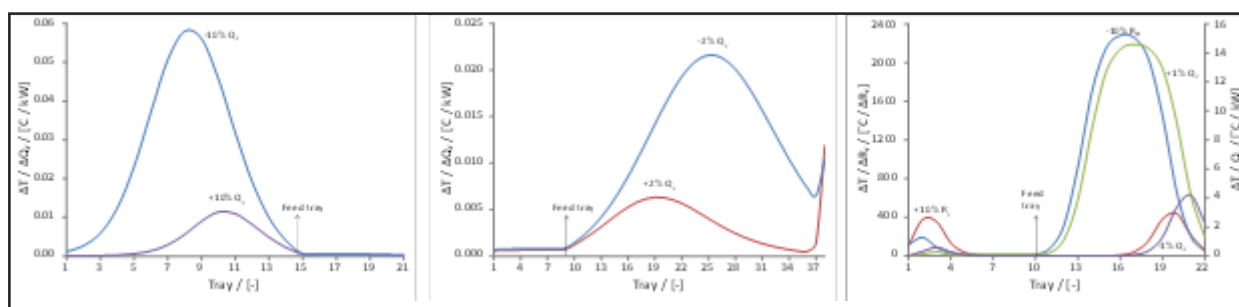


Fig. 5. Open loop steady state gain: column C1 (left), column C2 (middle), column C3 (right)

The *Slope criterion* could be also considered for avoiding locating the temperature controller in a zone with a flat temperature profile [10]. For dynamic reasons, it should be avoided locating a temperature sensor in a region with a small temperature change from one stage to the next because the initial dynamic temperature response is directly given by the difference in neighboring stage temperatures [10], and tight control would not be possible where the temperature changes are small. Large changes in temperature from tray to tray indicate a region where the compositions of important components are changing [9]. This criterion gives results similar to the sensitivity criterion.

According to figure 5 (left), tray 9 was chosen for temperature control in the first column.

In the second column, the loss of reactants from the system with the bottom product is indirectly prevented by bottom temperature control, adjusting the reboiler duty, while the reflux flow is maintained constant, on flow control. Looking to the temperature profiles (fig. 4, middle) and to the open loop steady state gains (fig. 5, middle), tray 28 is considered a sensitive tray and is selected for temperature control.

Dual-temperature control is required in the third column to prevent offspec alkylate product, which can be

contaminated with n-butane (heavy inert), and also alkylate loss with the overhead product, leading to profit loss. Trays 3 and 17 are selected for temperature control by manipulating the reflux flow and reboiler duty respectively.

### Flowsheet 2

In the second flowsheet (fig. 6) the separation section consists of two distillation columns: one for the separation of the propane, unconverted reactants and product, and the other one for the product purification. The propane (light inert) is removed from the system as vapor distillate, in the overhead stream of the first column, the reactants containing some n-butane (heavy inert) are recovered as a liquid side-stream and the products with the rest of n-butane in the column bottom. The control structure of Flowsheet 2 of the alkylation plant is also shown in figure 6.

The material balance of the steady state flowsheet corresponding to Flowsheet 2 is presented in table 2.

The reactors feed flowrates, temperature and inventory control is achieved similar to Flowsheet 1: the reactor inlet flowrates are set on flow control (fresh butene stream 2 and combined fresh isobutane and the recycle stream 1), reactors temperature is controlled with the reactor duty and the reactors inventory is controlled with the reactor effluent flow.

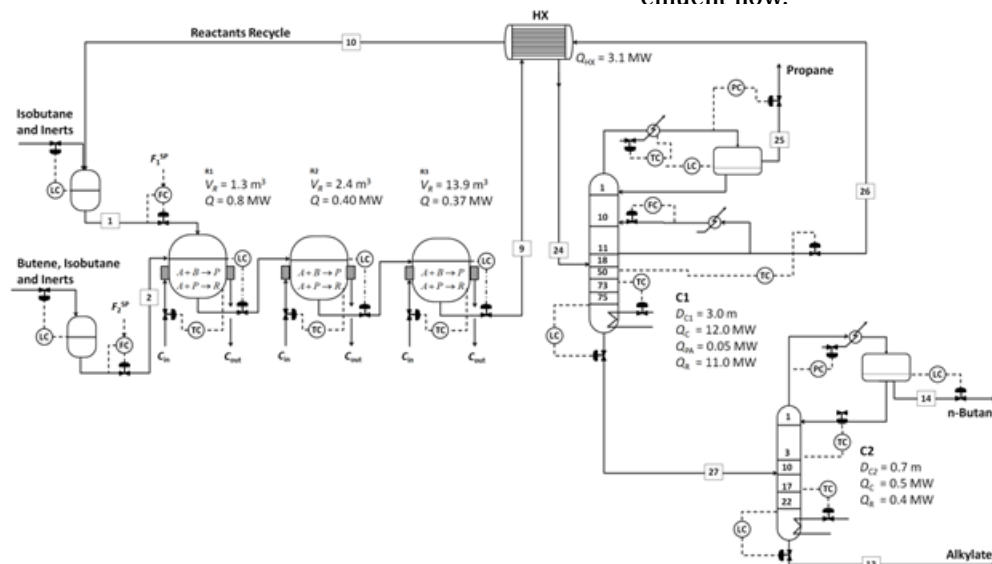


Fig. 6. Control structure - Flowsheet 2

Table 2  
FLOWSHEET 2 - STEADY STATE MATERIAL BALANCE

	Streams									
	1	F <sub>2</sub>	9	10	13	14	24	25	26	27
<b>Mole Flow [kmol/h]</b>										
Butene	0.0	52.5	3.1	3.1	0.0	0.0	3.1	0.0	3.1	0.0
Isobutane	29.0	21.0	1832.8	1832.5	0.0	0.1	1832.8	0.2	1832.5	0.1
Isooctane	0.0	0.0	47.3	0.4	46.1	0.8	47.3	0.0	0.4	46.9
Dodecane	0.0	0.0	2.8	0.0	2.8	0.0	2.8	0.0	0.0	2.8
Propane	3.1	5.4	26.9	18.4	0.0	0.0	26.9	8.5	18.4	0.0
n-Butane	27.9	27.0	228.7	173.9	0.0	54.9	228.7	0.0	173.9	54.9
<b>Total Flow [kmol/h]</b>	60	106	2142	2028	49	56	2142	9	2028	105
<b>Total Flow [kg/h]</b>	3444	5974	127060	117643	5736	3295	127060	387	117643	9031
<b>Temp. [°C]</b>	15.0	15.0	15.0	20.0	131.0	19.4	51.2	20.0	57.2	102.8
<b>Press. [bar]</b>	6.0	6.0	6.0	8.0	2.2	2.0	8.0	8.0	8.0	8.0
<b>Vapor fraction</b>	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.00	0.00	0.00

The second column (C2), for the product purification, is controlled similarly to the third column (C3) from Flowsheet 1, providing: condenser pressure control manipulating in reverse action the condenser duty; reflux drum and bottom level control manipulating in a direct action the product outlet flows (LV-configuration); and dual temperature control in the top and bottom sections of the column, for indirect composition control. The top temperature controller manipulates in direct action the reflux flow and the bottom one manipulates in reverse action the reboiler duty. The location of the temperature sensors is based on figure 4 (right) and fig. 5 (right).

The difference between Flowsheet 1 and Flowsheet 2 is made by the first column (C1) of Flowsheet 2, which merges the service of the first two columns (C1 and C2) from Flowsheet 1.

The control of the first column, with three product streams, is explained below.

The pressure of the column is controlled in a direct action by the amount of vapor released from the condenser. When the pressure increases, more vapors are removed from the column, and vice-versa. In order to prevent reactants carryover in the overhead product stream, the temperature in the condenser is controlled in reverse action by the condenser duty (indirect composition control). Since there is no liquid distillate product, the reflux drum inventory is controlled in direct action by the reflux flow.

The bottom part of the column has a straightforward control structure: the sump level control is performed by manipulating the product flowrate, and to avoid reactant loss in the product stream, tray temperature control is provided on tray 74, manipulating in reverse action the reboiler duty. Control tray selection is also based on *sensitivity* and *slope* criteria, considering figure 7 and figure 8.

A critical part for the stability of the second flowsheet is the control of the first column mid-section and more specifically, of the side-stream composition. Accumulation of n-butane in the recycle stream should be avoided because it could hamper the addition of fresh isobutane into the system. Lack of fresh isobutane feed leads to different reactant ratios in the reactors and variation of

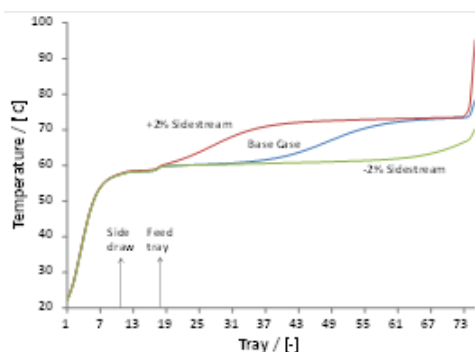


Fig. 7. Flowsheet 2 - base case and perturbed temperature profiles (Column C1)

selectivity towards the desired product. Therefore, it was decided to indirectly control the composition of the side-stream by controlling the temperature on a certain tray in the column.

The sensitivity analysis presented in figure 8, indicates that the proper trays to install the temperature controller are between 35 and 65, where the peaks of the open loop steady-state gain, for different changes in the manipulated variable (e.g. side-stream flowrate), are situated. Therefore, tray 50 was selected for installing the temperature sensor. The temperature controller works in reverse action, with the side stream flow as manipulated variable.

During the steady state simulation, a pump-around was provided on the draw-off tray to maintain the amount of liquid traffic into the column for effective fractionation. It slightly decreased the size and duty of the overhead condenser. Since it proved minor dynamic effects it was decided to keep its duty fixed and not using it for controlling the fractionation.

The control structure of the column is presented in figure 9.

The evaluation of the control structure provided satisfactory results with the plant running inside of its

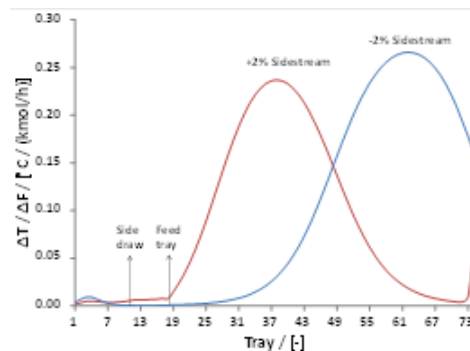


Fig. 8. Flowsheet 2 - Column 1 open loop steady state gain

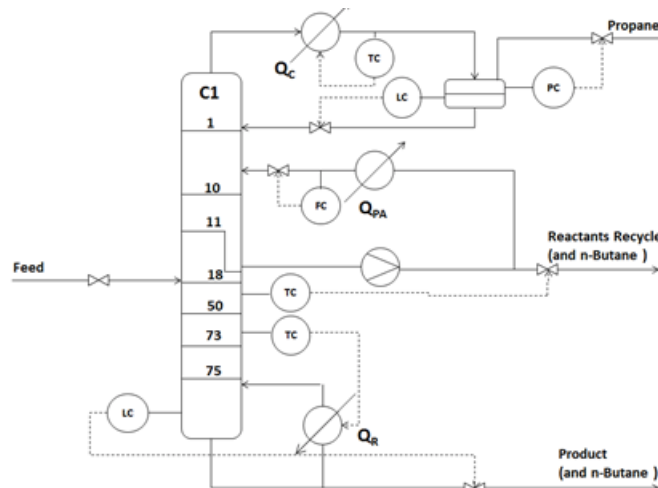


Fig. 9. Flowsheet 2 - control structure of the three-product column C1

	C1, Flowsheet 1	C2, Flowsheet 1	C1, Flowsheet 2
Tray # (theoretical)	23	38	75
Diameter, m	2.1	3.5	3.0
Reflux ratio	25.8	0.65	270.3
Cooling duty, MW	1.0	15.0	12.0
Reboiler / heating duty, MW	3.1	14.0	11.0
Condenser area, m <sup>2</sup>	96	563	1413
Reboiler area, m <sup>2</sup>	28	160	145
OPEX, \$/yr.	823,500	5,093,858	3,826,000
CAPEX, \$/yr.	218,900	692,000	1,093,000
TOTAL, \$/yr.	1,045,000	5,786,000	4,920,000

Table 3  
ECONOMICAL EVALUATION, BASED ON STEADY-STATE RESULTS

**Table 4**  
**FLWSHEET 1 - CONTROLLER PARAMETERS**

	Column 1		Column 2		Column 3			Reactors
	TC	PC	TC	PC	TC	TC	PC	TC
<b>Controlled variable</b>	Tray #9 temperature	Column pressure	Tray #28 temperature	Column pressure	Tray #3 temperature	Tray 17 temperature	Column pressure	Reactor temperature
<b>Controlled variable SP &amp; range</b>	28 °C 8 ÷ 48 °C	9.0 bar 8.5 ÷ 9.5 bar	58 °C 38 ÷ 78 °C	6.0 bar 5.5 ÷ 6.5 bar	55 °C 35 ÷ 75 °C	65 °C 45 ÷ 85 °C	2.4 bar 2.0 ÷ 2.8 bar	15 °C -5 ÷ 35 °C
<b>Manipulated variable</b>	Reflux flow	Condenser duty	Reboiler duty	Condenser duty	Reflux flow	Reboiler duty	Condenser duty	Reactor duty
<b>Manipulated variable nominal value &amp; range</b>	9900 kg/h 0 ÷ 20200 kg/h	-1.0 MW 0 ÷ -2 MW	14.0 MW 0 ÷ 28 MW	-15 MW 0 ÷ -30 MW	1120 kg/h 0 ÷ 2240 kg/h	0.5 MW 0 ÷ 1 MW	-0.45 MW 0 ÷ -0.9 MW	-0.8/-0.4/-0.4 MW
<b>Control action</b>	Direct	Reverse	Reverse	Reverse	Direct	Reverse	Reverse	Reverse
<b>K<sub>c</sub> (%/%)</b>	1	1	1	1	1	1	1	1
<b>T<sub>i</sub> (min)</b>	10	12	10	12	10	10	12	10
<b>Controller type</b>	PI	PI	PI	PI	PI	PI	PI	PI

operating window and according to the specifications. The accumulation of n-butane in the recycle stream was avoided and the variation of isobutane in the system could be compensated by the fresh isobutane. The dynamic response and the detailed results of the system to different disturbances are presented in section 4.

Cost estimation and a comparison between the two proposed flowsheets were performed. It was determined that the second flowsheet, with two columns, was with approximately 1,910,000 \$/yr. cheaper than the first flowsheet. The comparison considered only the process costs, neglecting the additional automation necessary for Flowsheet 1, which had one column more. The operating and investment prices for the two flowsheets are presented in table 3. The cost of the second flowsheet includes the price difference (~510,000 \$/yr.) between the feed-effluent heat exchangers (HX) in Flowsheet 1 and 2, downstream the reactors (at the inlet of the first columns, C1 and C1,2), due to the increased duty in Flowsheet 2 (higher temperature from the column). Details regarding the calculation of the costs can be found in [3].

### Controller tuning

#### Flowsheet 1

The algorithms used for the controllers within the alkylation unit are P and PI types. The controller parameters, gain and reset time, should be determined in order to achieve the proper control.

The rules of thumb provided in [7], indicate that an initial proper value for the controller gain ( $K_c$ ) would be 1 % / % (% OP range / % PV range). The reset time (integral time,  $T_i$ ) is recommended to be in the same range as the time constant of the process [7]. It is assumed that 10 min would be a suitable value for the time constant of a distillation process regarding the temperature control loop. For the control structure presented in figure 3, all controllers are tuned following this rule. The tuning parameters and the action of the controllers are shown in the table 4.

All level controllers, for the reflux drums, the sumps of the columns, the feed vessels and the reactors are P type controllers with a gain of 1 % / %. The ranges for the controlled and manipulated variables for level controllers were set to twice the nominal value.

#### Flowsheet 2

The tuning parameters of the reactor inlet flow controllers, temperatures and levels are identical to the ones presented in Flowsheet 1.

The pressure, temperature and level controllers of the first column are tuned following the same rules of thumb as previously mentioned [7]. Thus, all temperature controllers have a gain of 1 % / %. For the temperature controllers, dynamic simulations showed that increasing the reset times ( $T_i$ ) from 10 to 20 min provides a shorter time for reaching a new steady state and a smoother transient response of the temperature controllers. The temperature controllers with larger reset times behave less aggressive compared to the case of smaller reset times, where for a small process variable (PV) deviation, the controller immediately responds with a higher output (OP). The difference between the responses of the temperature controllers for the two alternatives (reset times of 20 min versus 10 min) is presented in figure 10.

The level controllers are all P type, while the temperature and pressure controllers are PI types.

The final parameters of the controllers installed for keeping the plant within its operating window, are presented in table 5.

### Plantwide dynamic response

The dynamic behavior and flexibility of the plant were assessed by considering different feed flowrates. Since there are two plant inlet feed streams, the change in flowrate of one of the two streams is also equivalent with a change of ratios between the components at the plant inlet. In previous studies of a similar reactor - separation - recycle system [12], it was observed the difficulty of maintaining the product specifications when changes of composition at plant inlet were considered. Table 6 presents the fresh feeds components molar flows and fractions before and after a + / - 10% flow disturbance in the fresh butene stream ( $F_2$ ).

In the following sections, the dynamic behavior of each flowsheet is discussed.

#### Flowsheet 1

The first flowsheet is subject to flow disturbances of the inlet butene stream  $F_2$ , in order to observe the dynamics of the plant and the response of the control system.

**Table 5**  
**FLOWSHEET 2- MAIN CONTROLLER PARAMETERS**

Controller type	Column 1				Column 2		Reactors	
	PC	TC	TC	TC	TC	TC	PC	TC
Controlled variable	Condenser pressure	Condenser temperature	Tray #50 temperature	Tray #73 temperature	Tray #3 temperature	Tray 17 temperature	Column pressure	Reactor temperature
Controlled variable SP & range	8 bar 7.5 ÷ 8.5 bar	20 °C 0 ÷ 40 °C	67 °C 47 ÷ 87 °C	74 °C 54 ÷ 94 °C	63 °C 43 ÷ 83 °C	129 °C 110 ÷ 150 °C	2.0 1.5 ÷ 2.5 bar	15 °C -5 ÷ 35 °C
Manipulated variable	Condenser vapor flow	Condenser duty	Side-stream flow	Reboiler duty	Reflux flow	Reboiler duty	Condenser duty	Reactor duty
Manipulated variable nominal value & range	8.7 kmol/h 0 ÷ 500 kmol/h	-12 MW 0 ÷ -25 MW	1.15 × 10 <sup>5</sup> kg/h 0 ÷ 2.3 × 10 <sup>5</sup> kg/h	11 MW 0 ÷ 22 MW	1136 kg/h 0 ÷ 2272 kg/h	0.4 MW 0 ÷ 0.8 MW	-0.5 MW 0 ÷ -1.0 MW	-0.8/-0.4/-0.4 MW
Control action	Direct	Reverse	Reverse	Reverse	Direct	Reverse	Reverse	Reverse
K <sub>c</sub> (%/%)	1	1	1	1	1	1	1	1
T <sub>i</sub> (min)	12	20	20	20	20	20	12	10
Controller type	PI	PI	PI	PI	PI	PI	PI	PI

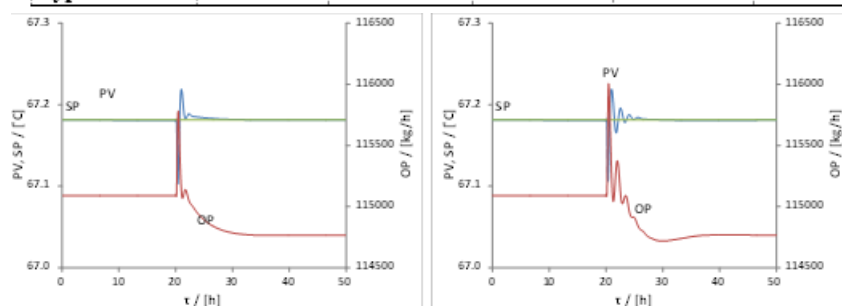


Fig. 10. Flowsheet 2 - C1 mid-section temperature controller (TC50) response  $T_i = 20$  min (left) and  $T_i = 10$  min (right).

Component	Mole fraction [mol/mol]		Mole flow [kmol/h]			
	$F_1$	$F_2$	$F_1$	$F_2$	$F_2, +10\%$ (disturbance 1)	$F_2, -10\%$ (disturbance 2)
Butene (B)	0.000	0.496	0.0	52.5	57.8	47.3
Isooctane (O)	0.000	0.000	0.0	0.0	0.0	0.0
Propane (C3i)	0.052	0.051	3.1	5.4	5.9	4.9
n-Butane (C4i)	0.465	0.255	27.9	27.0	29.7	24.3
Isobutane (iC4)	0.483	0.198	29.0	21.0	23.1	18.9
Dodecane (D)	0.000	0.000	0.0	0.0	0.0	0.0
<b>Total</b>	<b>1.000</b>	<b>1.000</b>	<b>60.0</b>	<b>105.9</b>	<b>116.5</b>	<b>95.4</b>

**Table 6**  
**FEED STREAMS MOLAR FLOWS AND FRACTIONS**

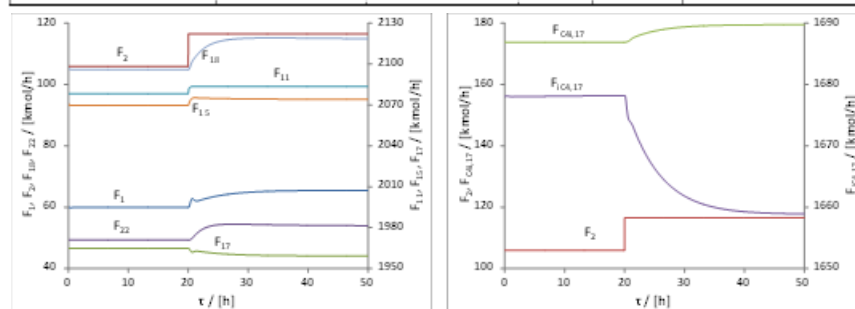


Fig. 11. Flowsheet 1 / Disturbance 1-stream flow rates (left) and recycled n-butane and isobutane flow rates (right)

Starting from the nominal steady state, at  $t = 20$  h, the butene feed flow rate  $F_2$  is increased by 10%. Results of dynamic simulation are presented in figure 11. The control system is able to bring the plant to a new steady state. When the butene flow is increased by 10%, the production also increases ( $F_{22}$  by 9.5%), while the recycle flow  $F_{17}$  slightly decreases (with 0.25%, 5 kmol/h); n-butane in the recycle, ( $F_{C4I17}$  right plot) increases because it is fed more into the plant as part of the butene feed stream. The decrease of the recycle flow  $F_{17}$  is due to increased

isobutane conversion when adding more butene into the system. The fresh isobutane stream,  $F_i$ , should therefore increase (9%) to reject the disturbance. An increase of 10% in the butene feed increases the isooctane production by 8.8%.

More butene fed into the system decreases the selectivity ( $\sigma$ ) because more butene is available for the secondary undesired reaction. The variation of selectivity was evaluated in reference to both the fresh butene amount

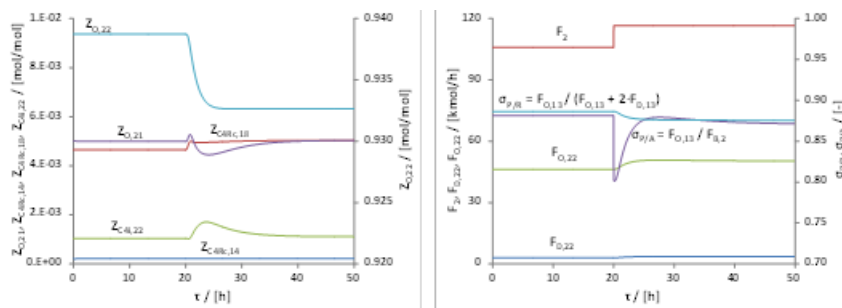


Fig. 12. Flowsheet 1 / Disturbance 1 - Compositions (left) and selectivity (right) changes

( $\sigma_{P/A}$ ) and to the dodecane by-product ( $\sigma_{P/R}$ ), as presented in figure 12 (right).

$$\sigma_{P/A} = \frac{F_{O,22}}{F_{B,2}} \quad (1)$$

$$\sigma_{P/R} = \left( \frac{F_{O,22}}{F_{O,22} + 2 \cdot F_{D,22}} \right) \quad (2)$$

Until now, the influence of the disturbance on the recycle flow, product flowrates and selectivity has been determined. Another important factor is to determine how product specifications can be met when the disturbance is applied and which separation control structure would work.

Variation of the butene feed stream leads to different yields and compositions of the streams within the plant and could hamper reaching the product specifications.

In theory, if the flowrates to a distillation process are all held as ratios and the temperature on any tray is held constant, all temperatures and compositions throughout the column should return to their original values. However, changing the ratio of components at plant inlet also changes the ratios at reactor outlet / column feed. In this case, the control of temperature on a tray cannot guarantee a constant composition, because in multicomponent systems the temperature does not correspond directly with certain ratio between the components [9]. As shown in fig. 12 (left), the impurity (n-butane,  $Z_{CAR,18}$ ) in the product stream  $F_{22}$  and the loss of reactants ( $Z_{CAR,18}$ ) varies with the step change in butene feed flow. However, the variations are small and the plant reaches soon a new steady state, which is almost insensitive to the disturbance. Therefore, it can be concluded that for an alkylation plant with this structure, the tray temperature control provides a satisfactory control of product specification.

The second disturbance applied to Flowsheet 1 involves feeding 10% less butene into the plant; therefore, starting from the nominal steady state, at  $t = 20$  h, the stream  $F_2$  is decreased by 10%.

The control system is also able to bring the plant to a new steady state (fig. 13, fig. 14). The effect is opposite to the previous case; when butene flow is decreased, the production ( $F_{22}$ ) also decreases, while the recycle flow  $F_{17}$  slightly increases. The recycle increases because, when less butene is added into the system, isobutane conversion decreases and it accumulates in the recycle stream. The fresh isobutane stream  $F_1$  decreases in order to reject the disturbance, keeping the reactor inlet flow ( $F_1$ ) constant and avoiding further accumulation in the recycle due to reactants imbalance. A decrease of 10% in the butene feed stream decreases the isooctane production by  $\approx 8.8\%$ .

Feeding less butene into the plant leads to a better selectivity towards isooctane (fig. 14 right), as less butene is available for the side-reaction.

The control structure proves to be able to maintain the plant within its operating window and in approximately 15 h, a new steady state is reached.

#### Flowsheet 2

In the second flowsheet there is only one column (C1) designed for the separation of the propane, as overhead vapor stream, reactants as side-stream and products in the bottom stream. The heavy inert (n-butane) is distributed between the side and the bottom streams.

A critical part for the stability of the second flowsheet is the control of the column (C1) mid-section, in order to maintain the n-butane concentration in the recycle within the design range. As described in 2.2, it is decided to indirectly control the side-stream composition with a temperature controller installed on tray 50, manipulating in reverse action the side-stream outlet flow. The proposed

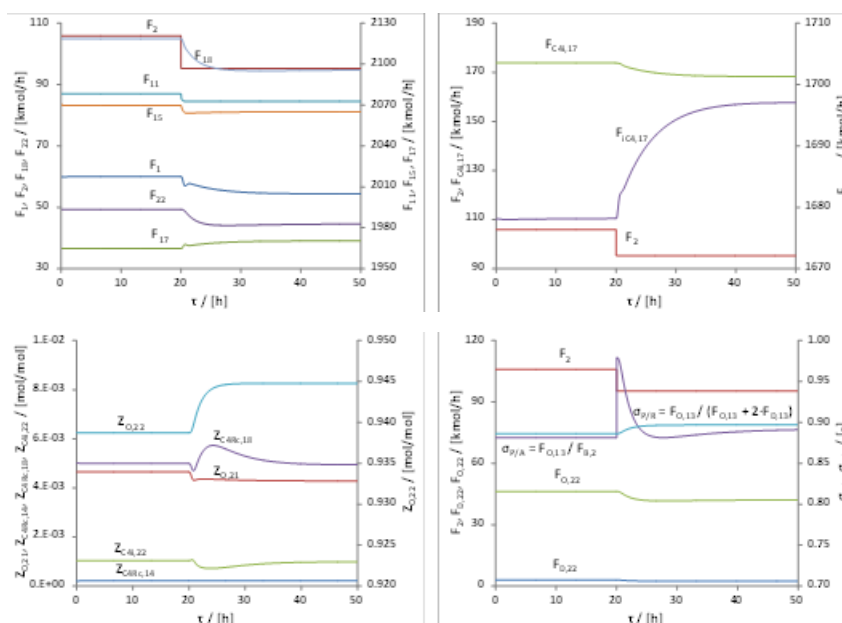


Fig. 13. Flowsheet 1 / Disturbance 2 - stream flow rates (left) and recycled n-butane and isobutane flow rates (right)

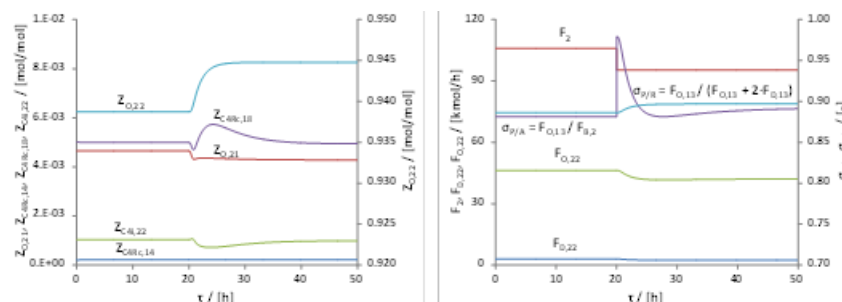


Fig. 14. Flowsheet 1 / Disturbance 2 - Compositions (left) and selectivity (right) changes



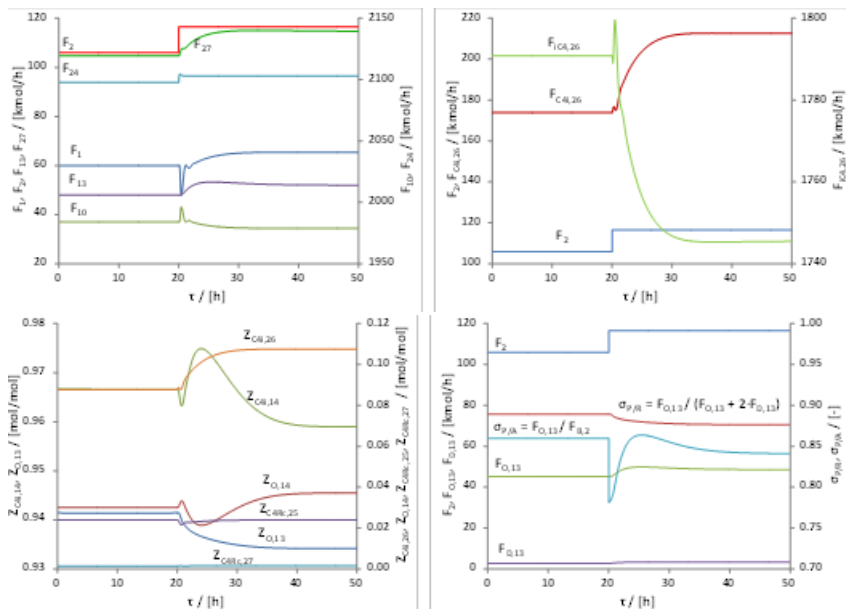


Fig. 15. Flowsheet 2 / Disturbance 1 - stream flow rates (left) and recycled n-butane and isobutane flow rates (right)

control structure, presented in 2.2, is challenged by applying a +/- 10% disturbance in the fresh butane flowrate  $F_2$ .

The dynamic response of the control system for a +10% disturbance in butene feed flow is presented in figure 15 and figure 16.

Similar behavior as with Flowsheet 1 can be observed; when 10% more butene is added into the system, the recycle stream  $F_{10}$  decreases, due to increased isobutane conversion, and the product flow  $F_{13}$  increases. Accumulation of n-butane in the recycle can be observed (fig. 15 right,  $F_{CA,26}$ ), as more is fed into the plant with the butene stream  $F_2$ .

The sensitivity analysis proved to be of utmost importance for the location of the temperature controller. The selection of tray 50 for locating the temperature sensor provides a stable operation of the column and of the entire plant, and a new steady state is reached in a relatively short time. The response of the temperature controller, when feeding 10% more butene, is presented in fig. 10 (left). When the temperature increases, the controller output (side-stream flowrate) decreases.

The second disturbance applied to this flowsheet involves feeding 10% less butene into the plant. Dynamic simulation results are presented in figure 17 and figure 18. The transient behavior of the system is similar to Flowsheet 1. The disturbance is followed by an increase in recycle

flow and less isobutane flow fed into the plant ( $F_1$ ). The production ( $F_{13}$ ) decreases by  $\approx 10\%$ , while the selectivity towards isooctane increases.

Accumulation of isobutane in the recycle stream, correlated with a temperature decrease on tray 50, can be observed in figure 17 (right) and figure 19.

The response of the temperature controller for a lower column feed flow ( $F_2$ ), given by less reactant feed into the plant, is presented in figure 19.

It is observed that, when less n-butane is fed into the plant (as part of  $F_2$  stream which is decreased by 10%), its concentration in the recycle (fig. 18,  $Z_{CA,26}$ ) and on tray 50, respectively, also decreases while the isobutane concentration increases; this leads to a slight decrease of temperature into the column, since n-butane is heavier than isobutane (NBP is -1 fC vs. -11.7 fC). The reverse action of the controller reflects the following: when the temperature on tray 50 decreases, the controller increases its output in order to increase the side-stream flow ( $S$ ). Considering the column overall material balance ( $F_{24} = F_{25} + F_{26} + F_{27}$ ), the top and bottom flowrates are decreased. Instead of removing n-butane via the bottom, it starts accumulating into the column and in the side-stream; this raises back the temperature on tray 50 until it reaches the set-point. Increasing the side-stream flowrate, the addition of fresh isobutane into the plant decreases (fig. 17 left,  $F_1$ ),

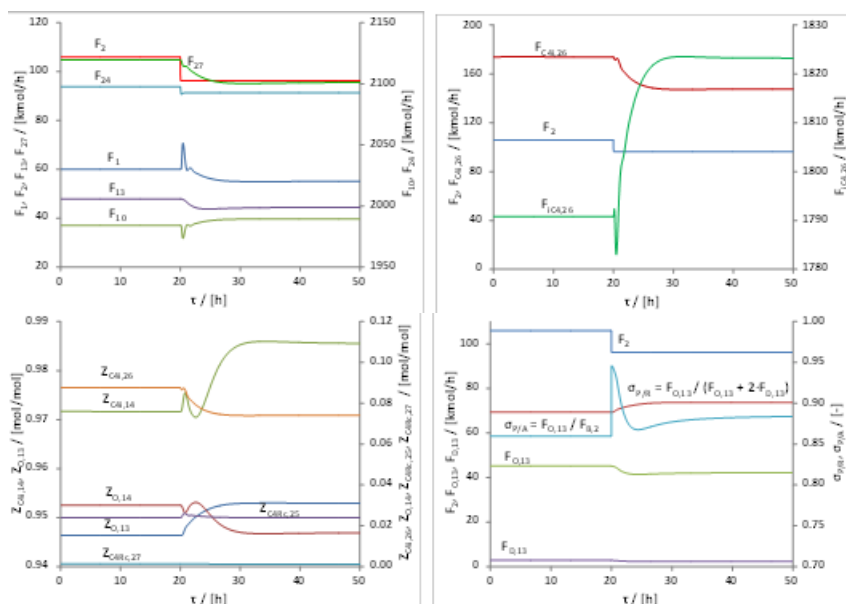


Fig. 16. Flowsheet 2 / Disturbance 1 - Compositions (left) and selectivity (right) changes

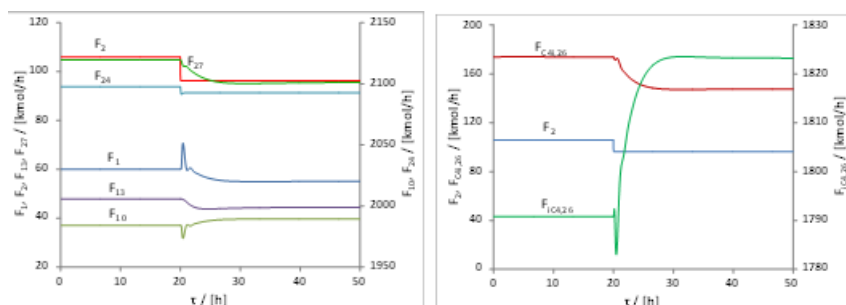


Fig. 17. Flowsheet 2 / Disturbance 2 - stream flow rates (left) and recycled n-butane and isobutane flow rates (right)

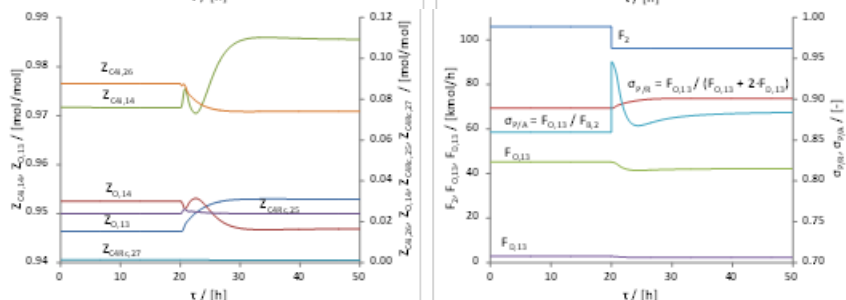


Fig. 18. Flowsheet 2 / Disturbance 2 - Compositions (left) and selectivity (right) changes

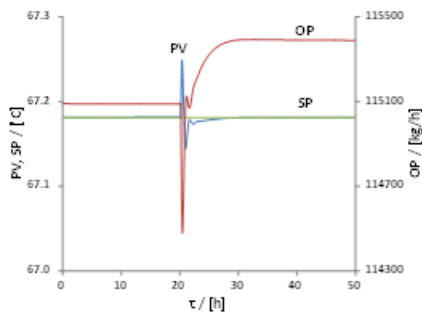


Fig. 19. Flowsheet 2 / disturbance 2 - C1 mid-section temperature controller (TC50) response

due to the set-up of the control structure; this also helps in decreasing the isobutane concentration in the column, favoring the increase of n-butane concentration.

## Conclusions

The transient behavior of the isobutane-butene alkylation process has been evaluated for different disturbances and two different flowsheets: with two and three separation columns for the product purification.

The performance of the plant had been previously evaluated via steady state simulations and sensitivity analysis [3, 5]. In this article, the flexibility of the steady state designs and the proposed control systems have been assessed by performing dynamic simulations. The change of the production rates and the control of impurities in the products were evaluated.

For both flowsheets, a production change by  $\pm 10\%$  could be achieved by a change of the fresh butene flowrate ( $F_2$ ) of similar magnitude. The selectivity towards isooctane varied inversely proportional with the fresh butene inlet flow.

A critical part for the stability of the second flowsheet was the control of the first column mid-section. The sensitivity analysis, for determining the location of the temperature controller / sensor, proved to be of outmost importance. Indirect control of the recycle stream composition, achieved by controlling the temperature on tray 50, avoided the n-butane accumulation in the recycle stream. Therefore, the addition of fresh isobutane was not hampered, when 10% more fresh butene mixture ( $F_1$ ) was added into the system.

Tray temperature control provided a good (indirect) composition control for all the columns; the variation of impurities in the products was insensitive to disturbances.

The evaluation of the control structures provided satisfactory results, with both plants running inside of their operating window and according to the specifications. Both flowsheets proved stability and flexibility, as the control structures were able to bring the plant to a new steady state each time a disturbance was applied.

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