

Case studies

The aim of this chapter is the description of the case studies that are contemplated in Chapter 7 (Results and discussion) and Chapter 8 (Industrial applications). Some interesting characteristics are commented in each case. First, the academic scenarios are presented, then the cases at pilot plant scale are described and finally the industrial scenarios are introduced.

6.1. Academic scenarios

Academic case studies correspond to test-bed problems published in the literature. They have been chosen taking into account the plant operation mode (continuous and batch) and their complexity (chemical processes involved).

6.1.1. *Plant with recycle*

The case of plant with recycle has been chosen to demonstrate the proposed method in a complex continuous chemical plant.

Inclusion of recycle streams in chemical processes sometimes has to be done to improve plant economics, in spite of the fact that from the point of view of process control it can seriously affect system performance making it more sluggish because the overall time constant is increased.

Information on control strategies for processes with recycle streams is relatively scarce when compared with control systems of unit operations working independently or in series where the effects of disturbances simply cascade from one unit to the next.

In addition, some control systems may fail in a plant-wide scenario while being effective for single unit operations. Interaction among control loops has also to be contemplated (Ruiz et al., 1999a). The evaluation of different control strategies can be performed efficiently by dynamic simulation (Basualdo et al., 2000).

An inventory control system, based on the fact that each whole process has an intrinsically self-regulating control structure, offers an improved and effective alternative strategy (Belanger and Luyben, 1997).

Process description

The plant has two main operating units: a reactor and a stripper (see Figure 6.1). Fresh feed, consisting of reactant *A* and some of product *P*, is fed to the reactor. The reactor is a continuous stirred tank. The irreversible reaction that occurs is of first order: $A \rightarrow P$. The reactor output is fed to the stripper. Most of unreacted *A* is separated from product *P* there. The plant's product, with a small mol fraction of *A* (X_{AB}) is obtained at the stripper's bottom. The stripper's output at the top is recycled to the reactor. The plant is designed around a fixed feed composition of $X_{A00} = 0.9$.

The physical properties of the components are the same except for the relative volatilities. The reaction rate law is $r = VR \times k \times X_{AF}$, where r is the rate of reaction, VR is the holdup of the reactor, k is the reaction rate constant and X_{AF} is the mol fraction of *A* in the reactor. The assumptions of the stripper's model are the following: constant pressure, constant molar flows, no vapor holdup, linear liquid dynamics, equilibrium on all stages, partial reboiler, total condenser, binary mixture, theoretical trays, saturated liquid flow feed and constant relative volatilities of $a_A = 2$, $a_B = 1$. It has 16 stages including reboiler and condenser.

The production rate is fixed at $B = 1.8 \text{ kg.mol. min}^{-1}$ at a desired purity $X_{AB} = 0.0120$. The reactor is run isothermally at a temperature that yields a specific reaction rate constant of $k = 0.00567 \text{ min}^{-1}$. The reactor effluent has a flowrate of $F \text{ kg.mol.min}^{-1}$ and a composition of X_{AF} . Liquid from accumulator is recycled to the reactor at a rate of $D \text{ kg.mol.min}^{-1}$ and a composition of X_{AD} . Table 6.1 summarises the steady-state design.

Control system

The inventory of the accumulator is controlled by the manipulation of the flowrate of the recycle stream. The inventory of the column base is controlled through the

manipulation of the bottom product flowrate. The cooling water flowrate to the condenser is used to control the pressure. Perfect control is assumed for this loop.

A Proportional-Integral controller manipulates the flowrate of steam to the reboiler of the stripper in order to regulate the purity of bottom's product. Equation (6.1) shows the calculus of the controller's output signal (CO), being E the error signal (difference between the set point and the process measurement), the $bias$ is a constant and is the value of the controller output when there is no error. The K_c is the controller gain and τ_i is the integral time.

$$CO = bias + K_U \cdot E + \frac{1}{t_i} \int E(t)dt \quad (6.1)$$

A 3-min deadtime is associated with the measurement of product composition. Settings were determined according to Tyreus and Luyben (1992). Equations (6.2) and (6.3) show the calculus for this loop, being P_u the ultimate period (min.) and K_u the ultimate process gain (%/min) in a relay-feedback test. By this way, the determined settings were: $K_c=0.1875$ and $\tau_i=29.58$ min.

$$K_c = \frac{K_u}{3.22} \quad (6.2)$$

$$t_i = 2.2 \cdot P_u \quad (6.3)$$

A proportional-only controller manipulates the flowrate of the reactor effluent stream in order to regulate reactor inventory. The K_c value selected is 5 (Belanger and Luyben, 1997).

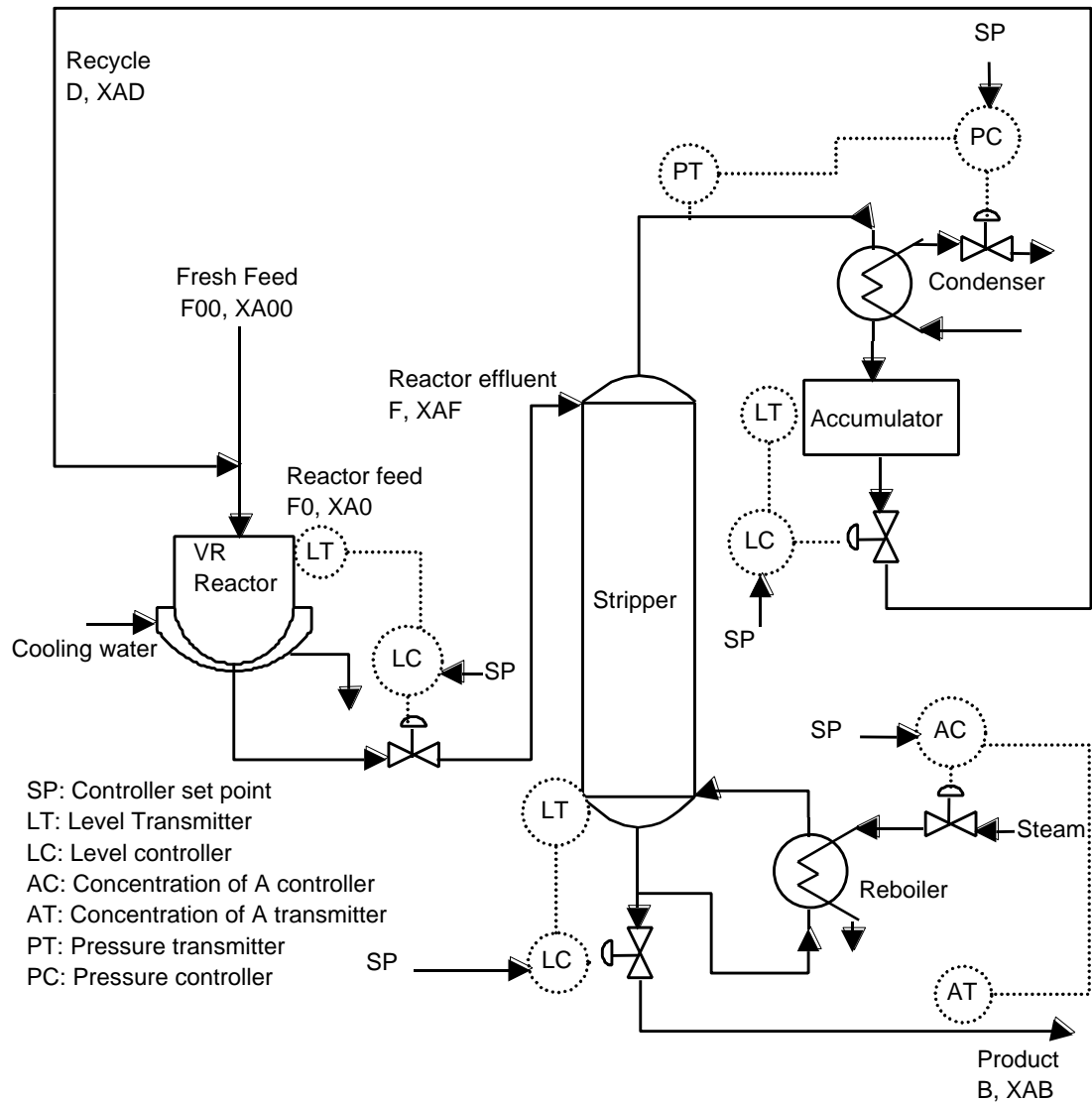


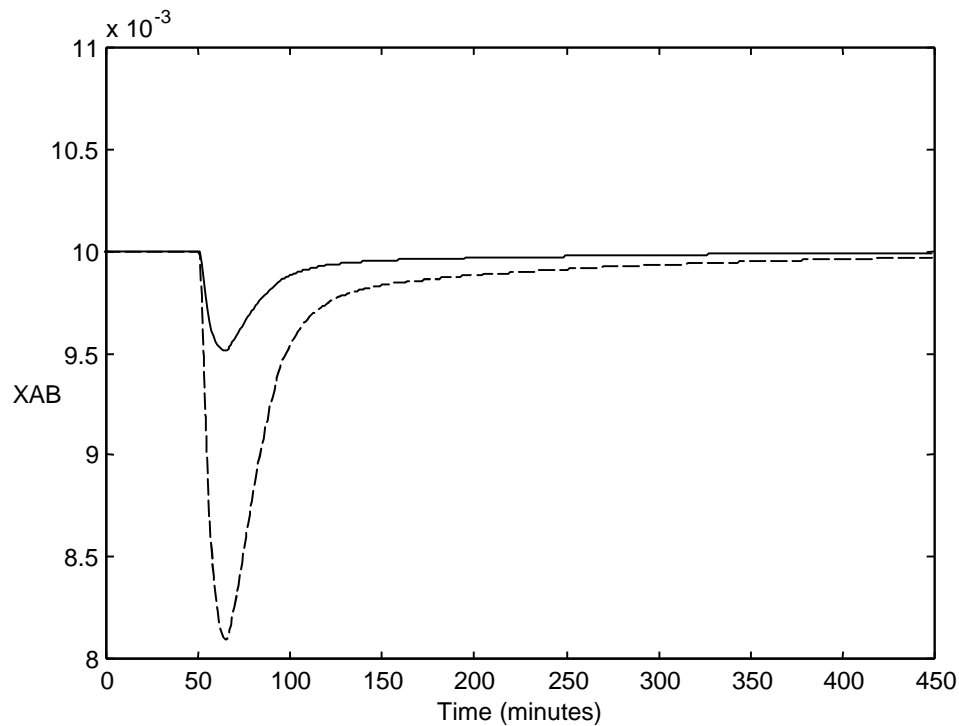
Figure 6.1. Chemical plant with a recycle stream

Table 6.1. Steady state design

F_{00} (kg.mol.min ⁻¹)	1.8	D (kg.mol.min ⁻¹)	4.3
X_{A00}	0.9	X_{AD}	0.543
F_0 (kg.mol.min ⁻¹)	6.98	B (kg.mol.min ⁻¹)	1.8
X_{A0}	0.65	X_{AB}	0.012
V (kg.mol)	750		

Features

The responses to the control actions are slow and also some faults are masked by the control system. As an example, when a pump failure or pipe leakage occur, the controlled bottom composition reaches its operating condition in a similar way to the case of a normal disturbance (e.g., small drop of the fresh feed flowrate, 5%) as can be seen in Figure 6.2 (normal and abnormal situation at time 50).



— Normal Disturbance

- - - Fault (pump failure or pipe leakage)

Figure 6.2. Masking knowledge by the control system

6.1.2. Batch reactor

The case study corresponds to a batch reactor introduced by Luyben (1990). It is shown in Figure 6.3. Reactant is charged into the vessel. Steam is fed into the jacket to bring the reaction mass up to a desired temperature. Then cooling water must be added to the jacket to remove the exothermic heat of reaction and to make the reactor temperature follow the prescribed temperature-time curve. This temperature profile is fed into the temperature controller as a set-point signal. The set-point varies with time. First-order consecutive reactions take place in the reactor as the time proceeds:

$A \rightarrow B \rightarrow C$

The desired product is component B.

In this work the Reaction monitoring is considered. The measurements are the steam valve openness, the water valve openness, reactor temperature, steam flowrate, water flowrate and the component A concentration (inferred).

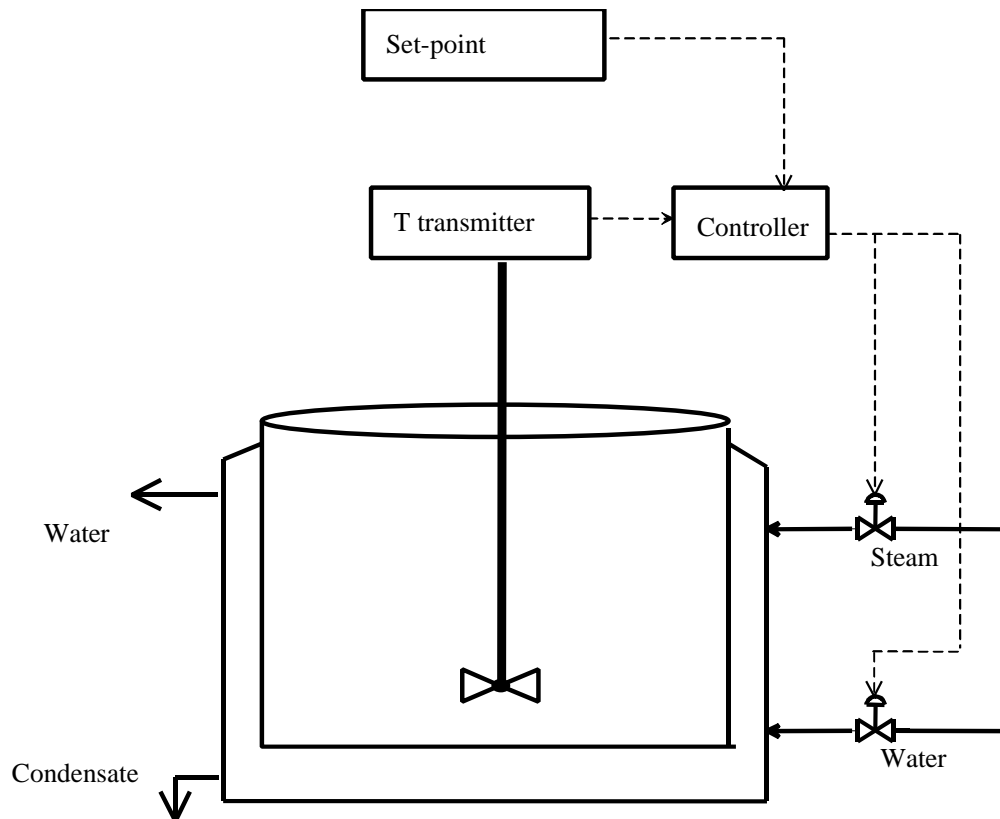


Figure 6.3. Batch reactor scheme

Figure 6.4 shows the profiles of the measured variables under normal operation conditions. The concentration of component B (not measured) has been also included.

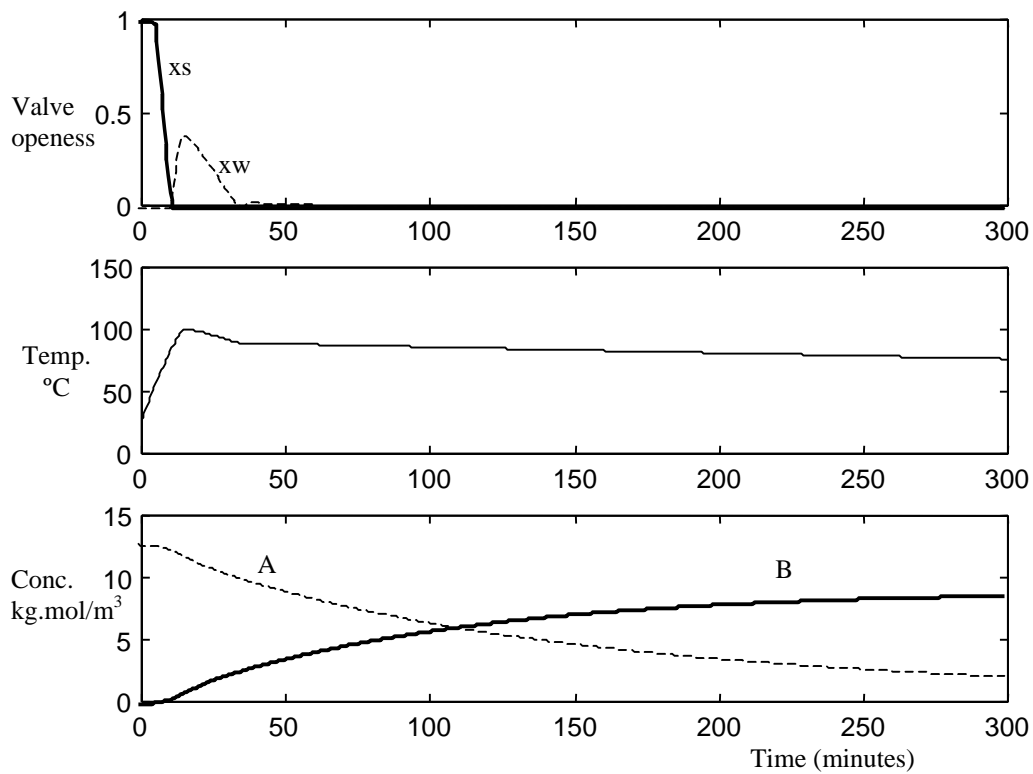


Figure 6.4. Profiles of measured and inferred variables under normal operating conditions

6.2. Pilot Plant scenarios

Scenarios at pilot plant scale, located at Universitat Politècnica de Catalunya (UPC), have been considered. The first one is being operating for several years and works in continuous mode, and historical data are available. The second one is a fed batch reactor and a validated model is available. Finally, the third scenario corresponds to a multipurpose batch chemical plants built at UPC that is at the stage of start-up, but simulations models are available.

6.2.1. Fluidised coal gasifier

Figure 6.5 shows a scheme of the pilot-scale coal gasifier unit (Nougués et al., 1999b). The fluidised bed reactor, made of ANSI-904L, operates at 930°C and 1.2 bar. Coal is fed through an electronically controlled rotary screw. The solid feed is introduced at the bottom of the reactor over the gas distributor. The gasifying agent, air plus steam, is fed at the reactor bottom side, allowing the solid fluidisation. Steam is produced by

adding water with a membrane pump. The desired product is the mixture of hydrogen plus carbon monoxide. Its production is measured indirectly by an on-line continuous monitoring and analysis of the carbon dioxide and oxygen in the output gases.

In Table 6.2, the references of sensors are presented. Historical data of this plant were used. Pressure measurements were not considered because there were not reliable data. Therefore, it is a scenario of diagnosis in presence of missing sensors.

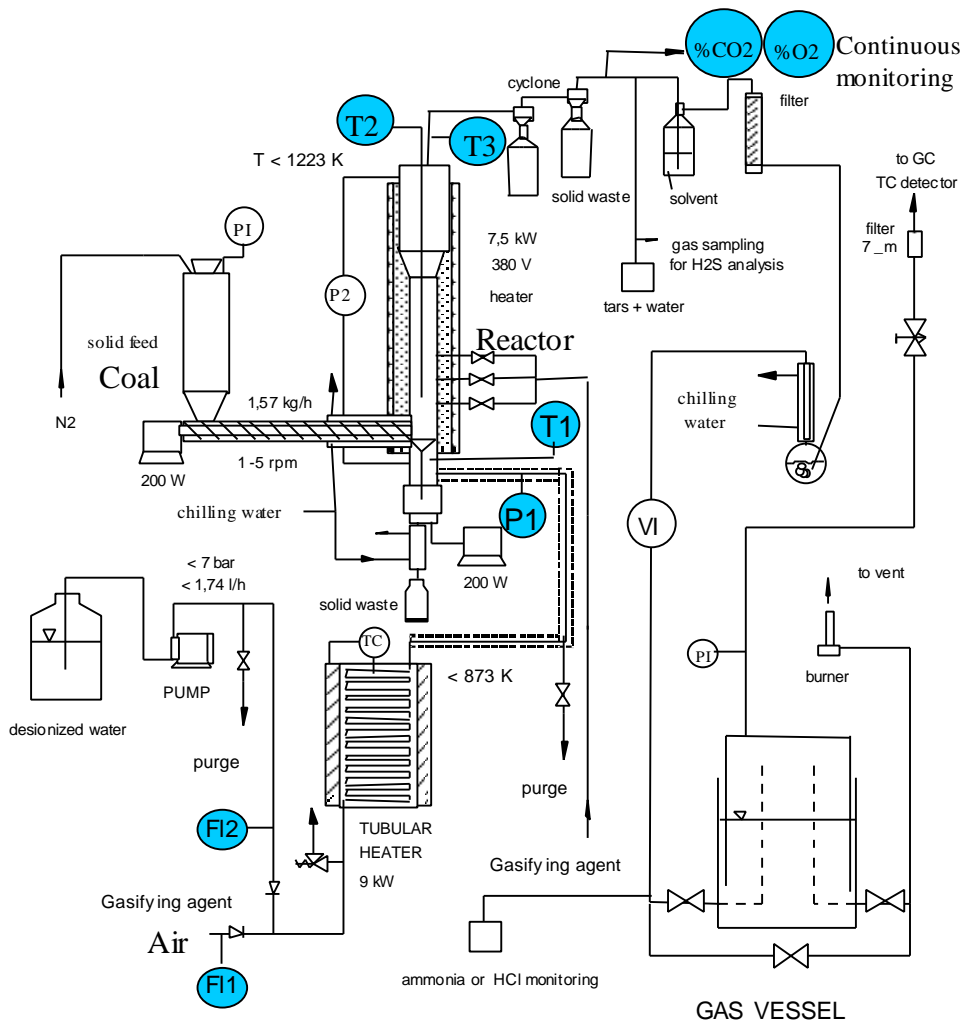


Figure 6.5. Fluidised bed Coal gasifier

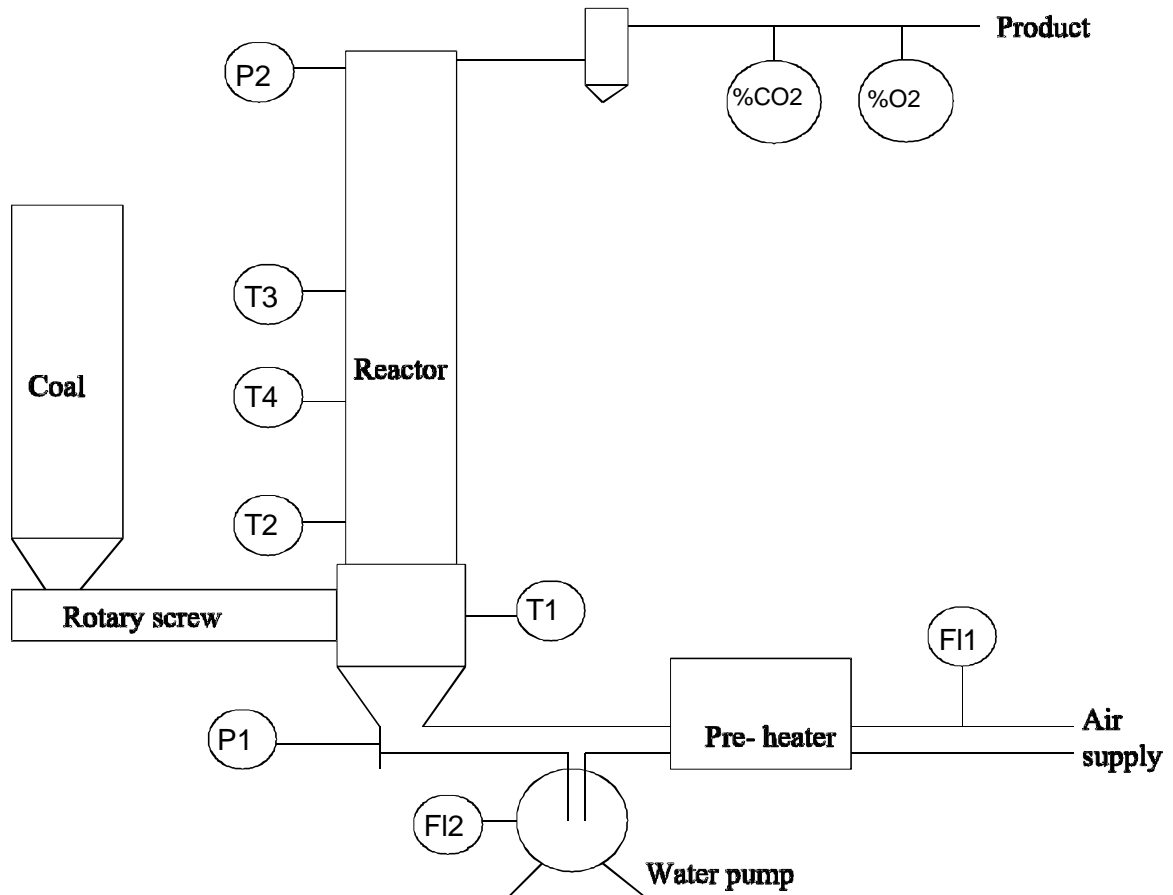


Figure 6.6. Simplified diagram of the monitoring system

Table 6.2. List of sensors in the pilot plant. Fluidised bed coal gasifier

Sensor	Variable measured
F1	Air flowrate
F2	Water flowrate
P1	Pressure (inlet of gases)
P2	Differential pressure
T1	Temperature in the inlet of steam and air
T2	Temperature in the base of reactor
T3	Reactor temperature
T4	Reactor external temperature
%CO ₂	%CO ₂ in the gas product
%O ₂	%O ₂ in the gas product

6.2.2. Fed-batch reactor

Figure 6.7 shows the flowsheet of the analysed fed batch reactor as it appears to the user via the programmed interface using Lab Windows CVI®. The reaction used has been the oxidation of $\text{Na}_2\text{S}_2\text{O}_3$ (sodium thiosulfate solution) with H_2O_2 (hydrogen peroxide). This is a well-known exothermic reaction. The pilot scale reactor consists of a 5-liter tank reactor, with a data acquisition system based on General Purpose Interface Bus and PC software. As mentioned above the reactor was operated in fed-batch mode being the H_2O_2 fed into $\text{Na}_2\text{S}_2\text{O}_3$. All the software for on-line control was developed in C programming language and the system analysis and model parameters adjustment was made in Matlab® developed modules (Nougués et al, 1999a, Grau et al., 2000).

The step analysed is the following: from the mixer tank a constant flow of reactants is discharged to the continuous stirred tank reactor (CSTR) by opening the valve V1. The reaction takes place in the CSTR and it is refrigerated constantly by opening valve Vref.

The measured variables are the following: reactor temperature, mixer and reactor levels and the inferred concentrations of reactants A (H_2O_2) and B ($\text{Na}_2\text{S}_2\text{O}_3$).

Profiles of such variables are shown, for normal operating operation, in Figures 6.8, 6.9 and 6.10.

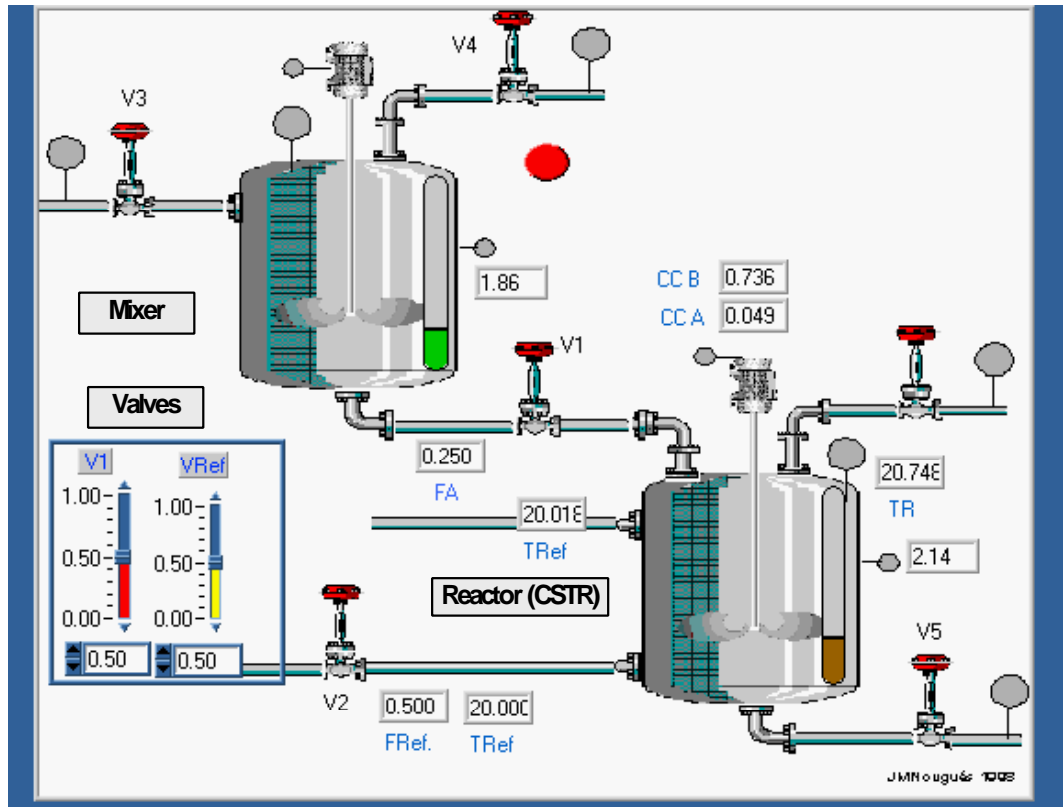


Figure 6.7. Scheme of the fed batch reactor as it appears in the user interface

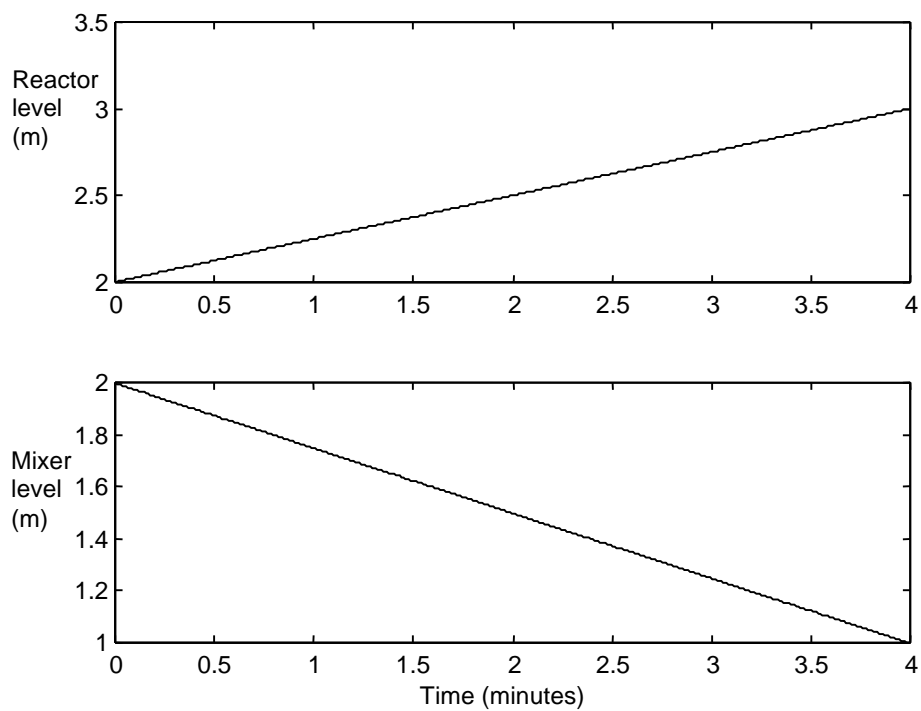


Figure 6.8. Profiles of tank levels during reactant A feeding

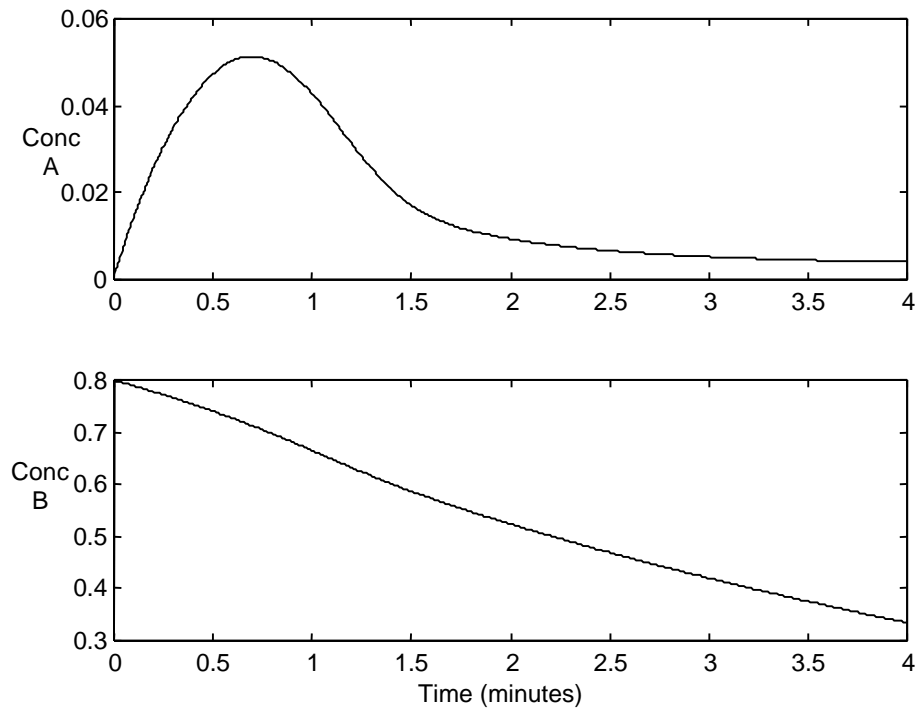


Figure 6.9. Profiles of inferred concentrations of reactants A (H_2O_2) and B ($Na_2S_2O_3$)

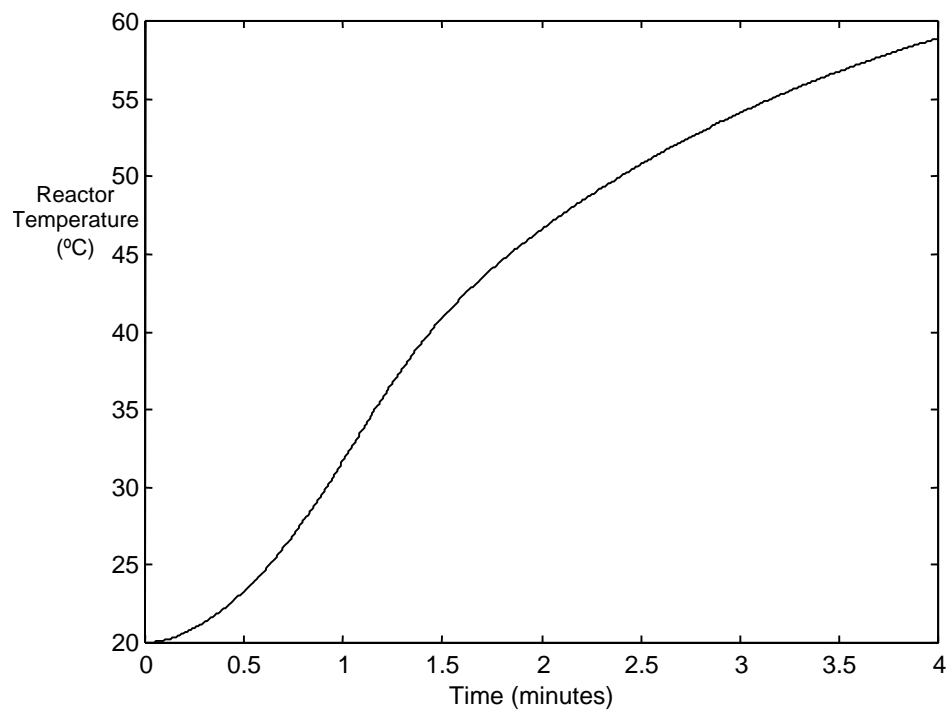


Figure 6.10. Profiles of direct and indirect measurements from the plant under normal operating conditions

6.2.3. Multipurpose batch chemical plant

Figure 6.11 shows the flowsheet of the multipurpose batch chemical plant considered for this case study. It has been built at UPC facilities and currently, the DCS is being programmed. A simulation of the actual configuration has been performed. The plant consists of three tank reactors, three heat exchangers and the necessary pumps and valves to allow changes of process configuration. Equipment of this plant is fully interconnected and the instrumentation allows configuration changes by software. Two recipes with two stages each one have been considered. Figure 6.12 shows the representation of recipes in a Gantt chart performing two batches. Table 6.3 shows the operation description and the operation times corresponding to the two recipes considered. Tank T1 is used to mix the reactants and then the mixture is discharged to the reactors R1 or R2 according to the schedule. Loading and discharge of tank T1 requires the same time for both recipes 1 and 2. On the other hand the time for homogenizing and stirring is longer for recipe 2. Furthermore, the time needed for reactor cleaning is different according to the recipe performed. In both recipes the times of the operation with code 5 (Reaction) are different depending on the reactor chosen to perform the second stage.

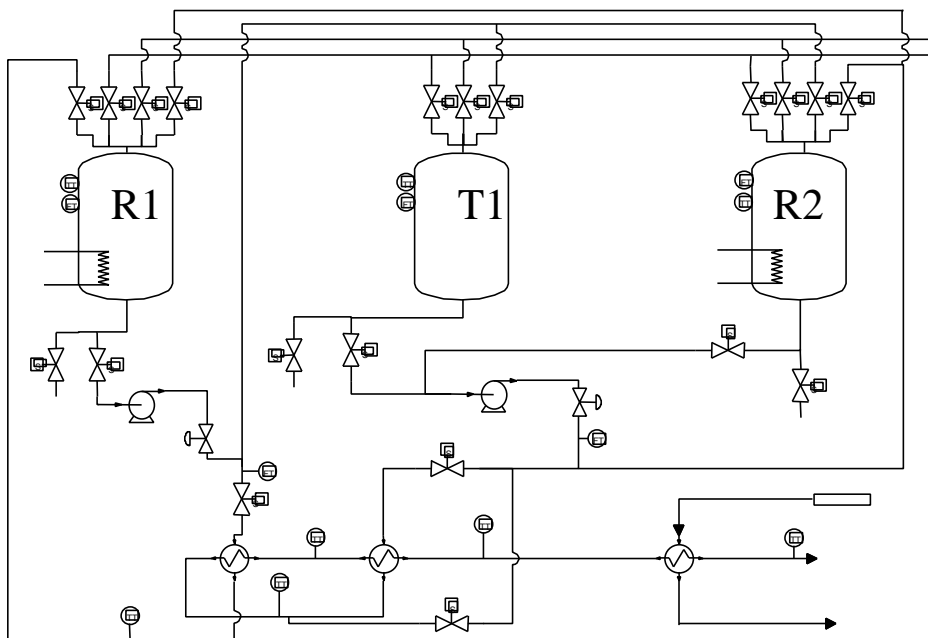


Figure 6.11. Flowsheet of the multipurpose chemical plant

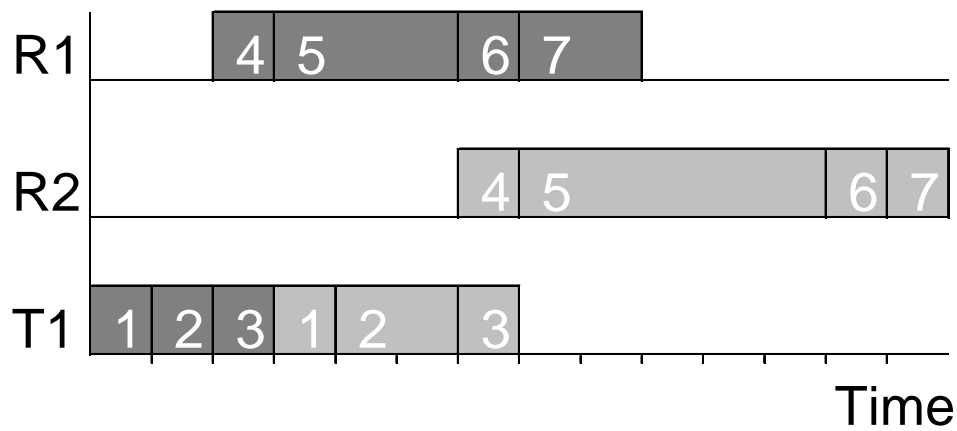


Figure 6.12. Gantt chart performing two batches.

Table 6.3. Operation description

Operation code	Stage code	Description	Unit	Recipe 1 (hours)	Recipe 2 (hours)
1	1	Load tank 1	T1	0.066	0.066
2	1	Stirring / Homogenising	T1	0.084	0.167
3	1	Discharge to R1 / R2	T1	0.066	0.066
4	2	Load reactor	R1/R2	0.066	0.066
5	2	Reaction	R1	0.25	0.33
5	2	Reaction	R2	0.33	0.416
6	2	Discharge of final product	R1/R2	0.066	0.066
7	2	Reactor cleaning	R1/R2	0.167	0.066

6.3. Industrial scenarios

Two Industrial cases correspond to sugar cane plants located in Latin America and involved in a European Research project coordinated by UPC (Project IC18-CT98-0271). The last industrial scenario correspond to a continuous real petrochemical plant.

6.3.1. Sugar cane refineries

Sugar industry involves a large amount of unit operations. From the point of view of the abnormal situation management there are two main problems. One of them is the strong interaction between the raw sugar plant and the refinery. The other one is the unavailability of some process variable measurements. The refinery presents a combination of steady state and batch processes. The process design and control are very complex (Crisafulli and Peirce, 1999). Therefore operators need a support for decision-making when a deviation from the normal operating conditions occurs (Alonso González et al., 1998).

Two plants are contemplated. One of the them has installed a DCS and the other one allows measurements in off-line mode.

CACSA Sugar refinery

Complejo Azucarera Concepción S.A. (CACSA) has a sugar plant located in Tucumán (Argentina). The refinery sugar process involves raw sugar dissolution, syrup treatment, boiling, crystallization, centrifuging and sugar drying, which are described below. This plant is in the same site of the raw sugar plant and has a capacity of 2000 ton per hour of refined sugar (Figure 6.13). There are three different sections as follows.

- Raw sugar dissolution and syrup treatment (continuous cycle): Raw sugar "I" is melted to form high purity syrup. This syrup is decolorized and filtered twice (using filter-press) before going to the syrup concentrator. Talo Floc system, char bone and ionic exchange resins are used to clarify the syrup. Syrup concentrator is three-effect evaporation system, heated by exhaust steam.
- Boiling, crystallization and centrifuging: A three-strike system, similar to raw sugar crystallization, is used for boiling syrup of high purity and low color. Sugar "A" from sugar pans "A" is of best quality Sugar "B" and "C" is mixed to obtain Common Refined Sugar. Refined sugar is only 85% of the saccharose sent to the refinery. The remaining 15% (refinery molasses "C") goes back to thick juice tank before raw sugar crystallization.
- Sugar drying: Rotative dryers are used for eliminating moisture and cooling sugar. Shaking screens are used to separate sugar grains bigger than the desired size.

Data from 1997 and 1998 champaigns are available. The refinery has the MODCELL acquisition data system. Table 6.4 shows the list of variables. Figure 6.14 shows a flow diagram of the refinery indicating the location of the available sensors.

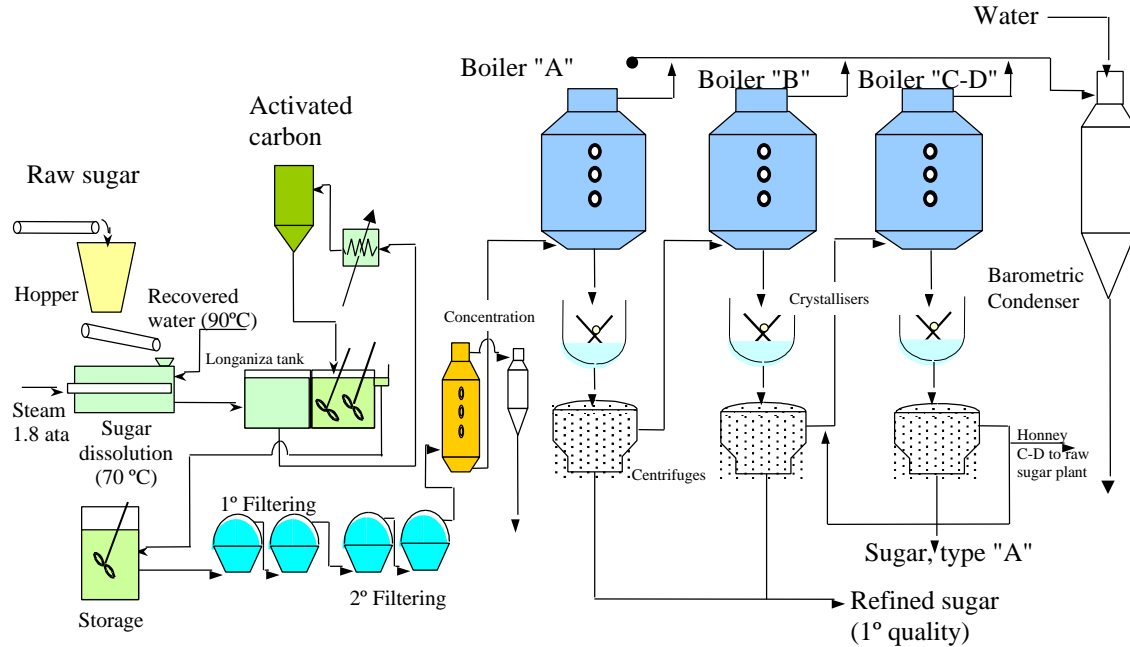


Figure 6.13. CACSA - Sugar cane refinery flowsheet

Table 6.4. Monitoring system

TAG	Description
TE-14	Syrup temperature in the dissolution station
FQ-13	Syrup flowrate to decoloration station
FT-26	Syrup flowrate in ionic exchangers
DIC-12	Syrup density
PI-16	Steam pressure
PI-17	Vaccum level
TIC-1	Dryer temperature
TIC-2	Dryer temperature
TE-03	Packaging temperature
TE-04	Packaging temperature
TE-05	Packaging temperature
TE-06	Packaging temperature
TE-07	Packaging temperature

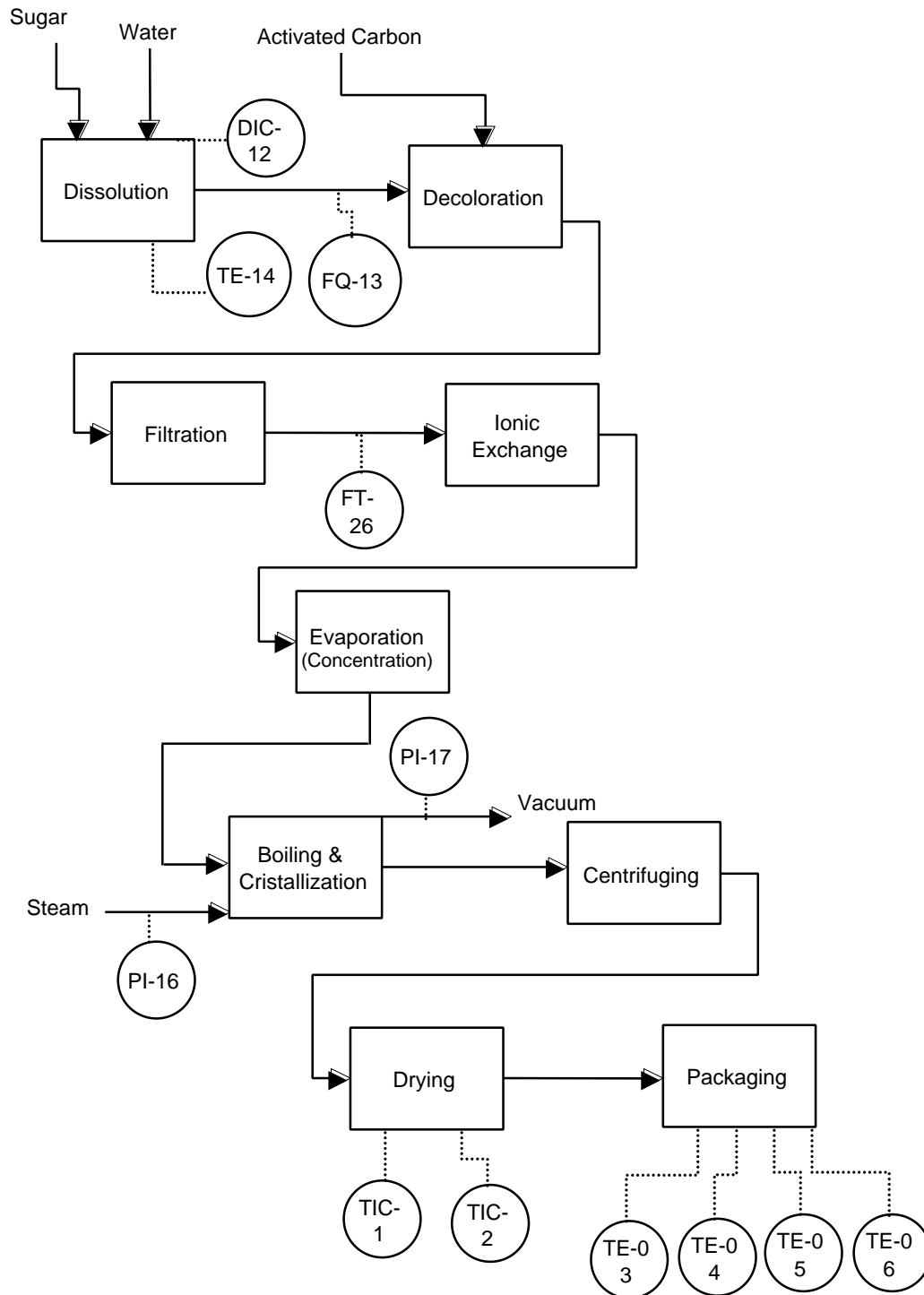


Figure 6.14. CACSA monitoring system

CAICC sugar refinery

A scheme of the refinery plant of Complejo Agroindustrial Azucarero Camilo Cienfuegos (CAICC) is shown in Figure 6.15. It correspond to the dissolution section.

A scheme of the dissolution (affination) plant of Complejo Agroindustrial Azucarero Camilo Cienfuegos (CAICC), located in Cuba, is shown in Figure 6.14. It corresponds to the plant dissolution section.

In the case of CAICC, the FDS has been developed based only on the HAZOP analysis and off-line measurements (there is not an on-line data acquisition system installed). A detailed flowsheet was developed and a complete HAZOP analysis of the preparation of the syrup for the refinery was carried out (see Annex A). The section was divided in four nodes:

- Raw sugar discharge to the mingler
- Raw sugar mingled
- Centrifugation
- Raw sugar dissolution

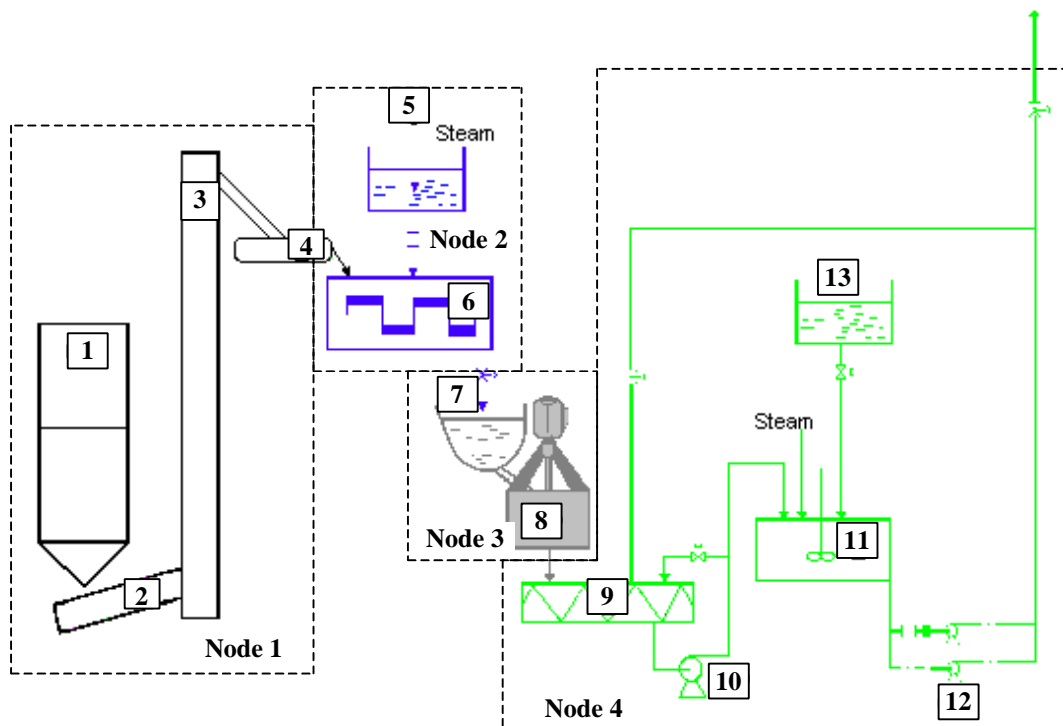


Figure 6.15. Scheme of a section of the Refinery plant (CAICC)

Table 6.5. Itemised description of the plant equipment and nodes

Ref	Equipment	Node
1	Sugar hopper	Raw sugar discharge to the mingler (Node 1)
2	Screw driver dispenser	
3	Elevator	
4	Weight of band	
5	Mingler	Raw sugar mingled (Node 2)
6	Refinery syrup tank	
7	Mixer of centrifuges	Centrifugation (Node 3)
8	Refinery centrifuge	
9	Pre- dissolutor	Raw sugar dissolution (Node 4)
10	Pump of the pre-dissolutor	
11	Dissolutor	
12	Crude liquor pump	
13	Water tank	

6.3.2. Petrochemical Plant

The case study corresponds to a real petrochemical plant (a section of "Complejo Lineal-Alquil-Benceno, Petroquímica La Plata -PLP", REPSOL-YPF, La Plata - Argentina-). It consists in a train of two distillation columns where a group of n-paraphines are separated from kerosene. Figure 6.16 shows the plant flowsheet taken from HYSYS.Plant simulation interface. Feed to the plant consists in a mix of hydrocarbons that is preheated in a heat exchanger which takes advantage of a lateral extraction of the second column (Re-distillation column). The light hydrocarbons (less than C-10) and Sulfur are separated in the first column (stripper) at the top (Light Kerosene). The stripper's bottom is fed to the Re-distillation column. At the top, the main product containing lineal hydrocarbons (C-10 to C-14) is obtained. At the bottom heavy kerosene is obtained as byproduct.

The interaction between the two columns is due to two facts: energy exchange between connecting flows and the linking of the stripper's bottom, which feeds the re-distillation column.

Historical plant data are also available (Plant Information system)

Further details of the process are withheld for commercial confidentiality reasons.

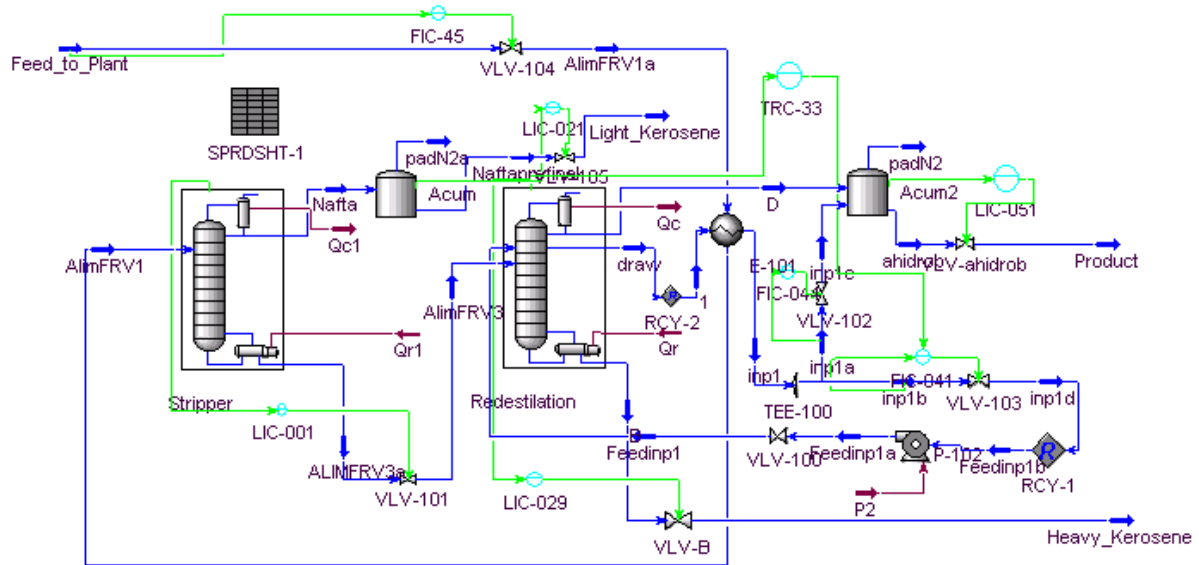


Figure 6.16. Plant flow sheet