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Dynamics and Control Strategies for a Butanol Fermentation Process

Adriano Pinto Mariano · Caliane Bastos Borba Costa · Maria Regina Wolf Maciel · Francisco Maugeri Filho · Daniel Ibraim Pires Atala · Dejanira de Franceschi de Angelis · Rubens Maciel Filho

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Abstract In this work, mathematical modeling was employed to assess the dynamic behavior of the flash fermentation process for the production of butanol. This process consists of three interconnected units as follows: fermentor, cell retention system (tangential microfiltration), and vacuum flash vessel (responsible for the continuous recovery of butanol from the broth). Based on the study of the dynamics of the process, suitable feedback control strategies [single input/single output (SISO) and multiple input/multiple output (MIMO)] were elaborated to deal with disturbances related to the process. The regulatory control consisted of keeping sugar and/or butanol concentrations in the fermentor constant in the face of disturbances in the feed substrate concentration. Another objective was the maintenance of the proper operation of the flash tank (maintenance of the thermodynamic equilibrium of the liquid and vapor phases) considering that oscillations in the temperature in the tank are expected. The servo control consisted of changes in concentration set points. The performance of an advanced controller, the dynamic matrix control, and the classical proportional-integral controller was evaluated. Both controllers were able to regulate the operating conditions in order to accommodate the perturbations with the lowest possible alterations in the process outputs. However, the performance of the PI controller was superior because it showed quicker responses without oscillations.

Keywords Flash fermentation · Biobutanol · Dynamics · Control · PI · DMC

A. P. Mariano (✉) · C. B. B. Costa · M. R. W. Maciel · R. M. Filho
Laboratory of Optimization, Design and Advanced Control, LOPCA School of Chemical Engineering, University of Campinas (UNICAMP), P.O. Box 6066, 13083-970 Campinas, São Paulo, Brazil
e-mail: adrianomariano@yahoo.com.br

F. M. Filho · D. I. P. Atala
Laboratory of Bioprocess Engineering, School of Food Engineering, University of Campinas (UNICAMP), Campinas, Brazil

D. d. F. de Angelis
Department of Biochemistry and Microbiology, Institute of Biosciences, São Paulo State University (UNESP), P.O. Box 199, 13506-900 Rio Claro, São Paulo, Brazil

Nomenclature

A	Dynamic matrix
f	Weighting factor
F_0	Fresh broth flow rate (m^3/h)
F	Fermentor outflow rate (m^3/h)
F_c	Flash tank inlet flow rate (m^3/h)
F_P	Permeate flow rate (m^3/h)
F_{PU}	Fermentor purge flow rate (m^3/h)
F_r	Flash tank liquid outlet flow rate (m^3/h)
F_{re}	Return stream flow rate (m^3/h)
F_V	Flash tank vapor outlet flow rate (m^3/h)
I	Identity matrix
k	Sampling time
K_c	Proportional gain
K_i	Equilibrium constant
n	Time n
N_C	Control horizon
N_P	Prediction horizon
P_{but}	Butanol concentration in the fermentor (g/L)
P_{flash}	Flash tank pressure (kPa)
P_i^{sat}	Vapor pressure of component i (kPa)
P_0	Inlet product concentration (g/L)
P_i	Fermentor product concentration (g/L)
P_{ri}	Concentration of product i in the flash tank liquid outlet flow (g/L)
P_v	Product concentration in the flash tank vapor outlet flow (g/L)
r_x	Rate of cell growth (g/L h)
r_s	Rate of substrate utilization (g/L h)
r_{P_i}	Rate of products production (g/L h)
S_0	Inlet substrate concentration (g/L)
S	Fermentor substrate concentration (g/L)
S_r	Substrate concentration in the flash tank liquid outlet flow (g/L)
S_v	Substrate concentration in the flash tank vapor outlet flow (g/L)
T_{ferm}	Fermentor temperature (°C)
T_{flash}	Flash tank temperature (°C)
V	Volume of the fermentor (m^3)
x_i	Liquid molar fraction of component i
X_0	Inlet biomass concentration (g/L)
X	Fermentor biomass concentration (g/L)
X_c	Biomass concentration in the flash tank inlet flow (g/L)
x_n	Controlled variable error (set point minus measured value)
X_P	Biomass concentration in the permeate (g/L)
X_r	Biomass concentration in the flash tank liquid outlet flow (g/L)
X_v	Biomass concentration in the flash tank vapor outlet flow (g/L)
y_i	Vapor molar fraction of component i
y_0^{meas}	Present measured value of the variable
$y_{OL,i}$	Value predicted by the convolution model
$y^{set\ point}$	Set point value
$(\Delta m_k)^{new}$	Vector of optimal values of future changes in the manipulated variables on the control horizon

$(\Delta m_k)^{old}$	Past changes in the manipulated variable
Δm_n	Variation of the manipulated variable
Δt	Sampling time
γ_i	Activity coefficient
τ_i	Integral gain

Introduction

The acetone–ethanol–butanol (ABE) fermentation, as the fermentation to produce butanol is normally called, has a long history as an industrially significant fermentation. During the first half of the last century, ABE plants become a major industrial fermentation process recognized as second only to yeast-based ethanol fermentation. However, after this period, the production of biobutanol on a commercial scale has been considered to be uneconomical due to increasing substrate cost and availability of much cheaper petrochemically derived butanol. In addition, butanol productivity is low due to product toxicity [1, 2].

Engineering techniques to ferment and remove product simultaneously so that a toxic butanol concentration inside the reactor is never reached have been investigated during the past two decades. Product-removal techniques include gas stripping, liquid–liquid extraction, membrane-based methods (pervaporation, perstraction, and reverse osmosis), and adsorption [2]. By lowering the effect of product inhibition, substrate concentration can be increased, which results in reduction in the process streams, higher productivity, and lower distillation costs [3].

The flash fermentation technology, which is the process considered in the present study, is another technique that has been investigated [4]. In this continuous process, the fermentor remains at atmospheric pressure while the broth is circulated to a flash tank where butanol is boiled off.

The operation of large-scale fermentation processes in a continuous mode is desirable, since higher productivity, lower production cost, and better process control are attained. However, the industrial implementation of a continuous process first requires a study on the process behavior and its use in the development of an efficient control strategy. In this sense, mathematical modeling is a powerful tool, since the many different operating conditions investigated via simulations would not have been economically feasible to undertake experimentally with a large-scale bioreactor.

The industrial implementation of the flash fermentation to produce butanol will demand a control strategy robust enough to deal with common fluctuations in the quality of the agricultural raw material. For example, in ethanol fermentation, one of the sources of raw material quality fluctuation is the alteration in the amounts of treated sugarcane juice and molasses used in the composition of the medium fed to the fermentor. These alterations are made due to the sugar factories' operation. Furthermore, molasses undergoes a variation of composition in different crops. Consequently, the control must be designed in order to keep the substrate conversion at a desired value and to avoid variations in butanol concentration in the fermentor.

Another point of concern about the operation of the flash technology is the maintenance of the conditions (pressure and temperature) of the flash tank in order to ensure the thermodynamic equilibrium between the liquid and vapor phases, since alterations of these conditions can break the equilibrium and thus interrupt the butanol recovery. This problem was observed experimentally by Atala [5] when developing the flash fermentation process, as presented here, for the ethanol fermentation.

In a previous work, Costa et al. [6] studied the control of the flash fermentation process applied to ethanol fermentation. The control algorithm used was the dynamic matrix control

(DMC), which was able to control substrate and product concentrations. Other examples of successful use of DMC in continuous fermentations include the studies reported by Goochee et al. [7], Silva et al. [8], and Lunelli et al. [9].

The DMC algorithm is robust [10], and due to its predictive characteristics, it generally leads to better results than the classical proportional-integral derivative (PID) control, as reported by Rodrigues and Maciel Filho [11] and Meiena et al. [12], when studying the control of a fed-batch bioreactor and a solid-state fermentation bioreactor, respectively. On the other hand, PID controllers have been the most frequently used control technique in chemical and biochemical processes due to historical factors and implementation facilities. Examples of studies on control of continuous fermentative processes by PID include Karim and Traugh [13], Valarmathi et al. [14], and Galluzzo et al. [15].

Thus, the aim of the present work was to employ mathematical modeling to analyze the dynamic behavior of the flash fermentation process so that the best control strategies can be chosen to deal with two problems: the fluctuation of the sugar concentration in the raw material and disturbances of the temperature in the flash tank. The performance of an advanced controller, the DMC, and the classical PI controller was assessed considering different control strategies.

Material and Methods

Flash Fermentation Process

Figure 1 presents a general scheme of the flash fermentation process. The process consists of three interlinked units: fermentor, cell retention system (tangential microfiltration), and vacuum flash tank. Before steady state is reached, the operation of the process is similar to that of a conventional continuous process. Once steady state is attained, the vacuum separation system is activated and a partial separation of the solvents and water mixture occurs while the fermentation broth is circulated through this separation system. Butanol concentrates in the vapor phase, which is combined after condensation with the purge and permeate streams, and then sent to distillation [4]. The liquid stream leaving the flash tank returns to the fermentor.

Mathematical Modeling

The process dynamics are described by mass balances given by Eqs. 1, 2, and 3, in which the fermentor volume is assumed to be constant. Kinetic parameters of the ABE fermentation were determined experimentally by Mulchandani and Volesky [16].

$$\frac{\mathrm{d}X}{\mathrm{d}t} = r_X - \frac{(F_{\mathrm{PU}} + F)}{V}X + \frac{F_r}{V}X_r + \frac{F_{\mathrm{re}}}{V}X_{\mathrm{c}} + \frac{F_0}{V}X_0 \tag{1}$$

$$\frac{\mathrm{d}S}{\mathrm{d}t} = r_{\mathrm{S}} - \frac{(F_{\mathrm{PU}} + F - F_{\mathrm{re}})}{V}S + \frac{F_r}{V}S_r + \frac{F_0}{V}S_0 \tag{2}$$

$$\frac{\mathrm{d}P_i}{\mathrm{d}t} = r_{P_i} - \frac{(F_{\mathrm{PU}} + F - F_{\mathrm{re}})}{V}P_i + \frac{F_r}{V}P_{r\ i} + \frac{F_0}{V}P_{0i} \tag{3}$$

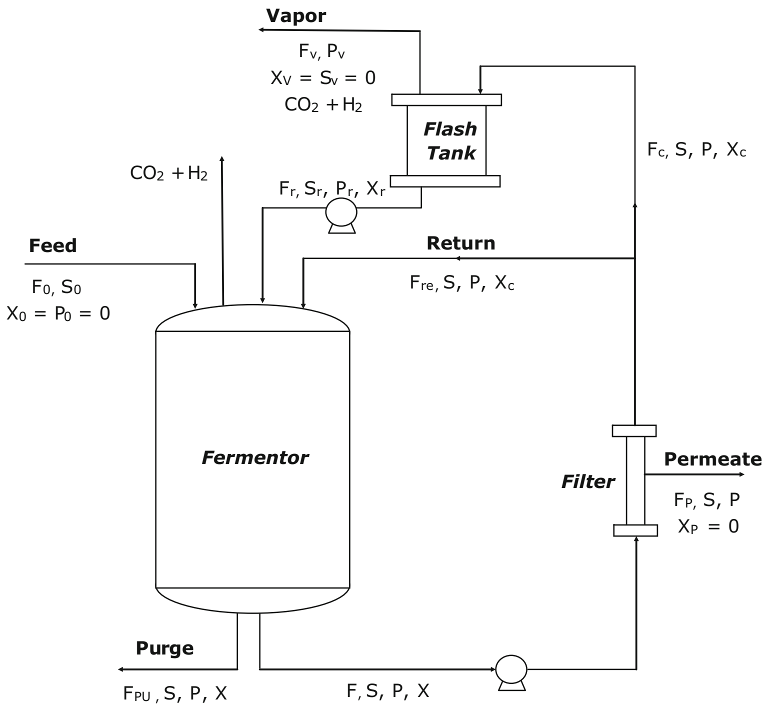

Fig. 1 General scheme of the continuous flash fermentation process

where i stands for butanol, acetone, ethanol, butyric acid, and acetic acid. The reader is referred to Mulchandani and Volesky [16] for details on the kinetic model equations (r_x, r_s, and r_{P_i}).

The mass balance of the flow streams (considering constant density) is given by Eqs. 4 and 5:

$$F_P = F_0 - F_{PU} - F_V \tag{4}$$

$$F = F_P + F_{re} + F_c \tag{5}$$

A "pseudo" steady state can be assumed for the flash tank since its dynamics are much faster than that of the fermentation process. Thus, the mass balance over the flash tank is given by Eq. 6.

$$F_c = F_V + F_r \tag{6}$$

The isothermal and isobaric evaporation model reported by Sandler [17] was used to describe the phase separation that takes place in the flash tank. The flash calculation consists in finding a solution to Eq. 7, which describes the vapor–liquid equilibrium. In this calculation, a multicomponent system (water, butanol, acetone, ethanol, acetic acid, and butyric acid) was considered, and P_i^{sat} was calculated by Antoine's equation and the activity coefficient (γ_ι) by the UNIQUAC model. The resulting set of equations Eqs. 6 and 7 was solved by the Newton–Raphson method.

$$K_i = \frac{y_i}{x_i} = \gamma_i \frac{P_i^{sat}}{P_{flash}} \tag{7}$$

where i stands for butanol, acetone, ethanol, butyric acid, and acetic acid.

Thus, the flash fermentation process was simulated by solving two deterministic models, one that represents the fermentor and the other for the flash tank. The equations that represent the dynamics of the fermentor (Eqs. 1, 2, and 3) in conjunction with the mass balances of the flow streams Eqs. 4 and 5 and the flash calculation Eqs. 6 and 7 were solved using a Fortran program. Integration of Eqs. 1, 2, and 3 was carried out by the fourth order Runge–Kutta method.

The concentration dynamics in the fermentor are highly altered when the flash tank starts to operate (Figs. 2 and 3). Once butanol is continuously recovered in the flash tank, its concentration in the fermentor lowers. This condition enhances cell growth and consequently substrate consumption. Before turning on the separation system, most of the glucose in the concentrated feed is not consumed, and for this reason, glucose concentration in the fermentor increased up to approximately 90 g/L. With the butanol recovery, biomass was able to increase (from 11.5 to 30.4 g/L) and consumed 95% of the glucose.

For the simulation presented in Figs. 2 and 3, optimized operating conditions were considered (Table 1) [18]. The magnitude of these values shows that the process was designed in order to produce butanol on an industrial-scale basis.

Process Control

Feedback control strategies were formulated in order to deal with regulatory and servo problems. In relation to the former, the objective of the control was to keep sugar and/or butanol concentrations in the fermentor constant in the face of disturbances in the feed substrate concentration. Another objective was the maintenance of the proper operation of the flash tank (maintenance of the thermodynamic equilibrium of the liquid and vapor phases) considering that oscillations in the temperature in the tank are expected. The servo control consisted in changes of concentration set points. Control loops [single input/single output (SISO) and multiple input/multiple output (MIMO)] were formulated based on the study of the dynamics of the process and are detailed in "Results and Discussion."

The performance of two controllers found in industrial-scale processes, the DMC and the PI controller, was assessed. An overview of the controllers is presented next.

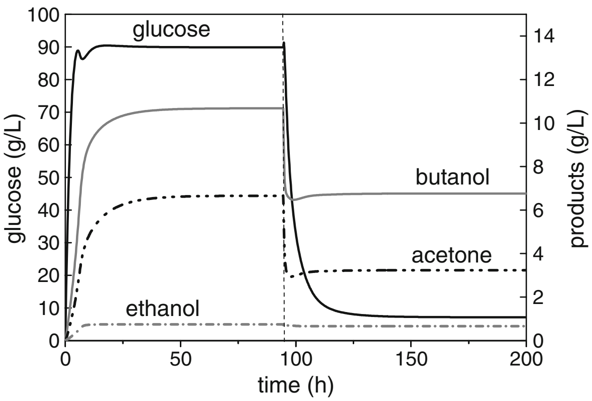

Fig. 2 Dynamics of glucose and products (butanol, acetone, and ethanol) concentrations in the fermentor. The *vertical dashed line* indicates the time when the flash tank separation system is turned on

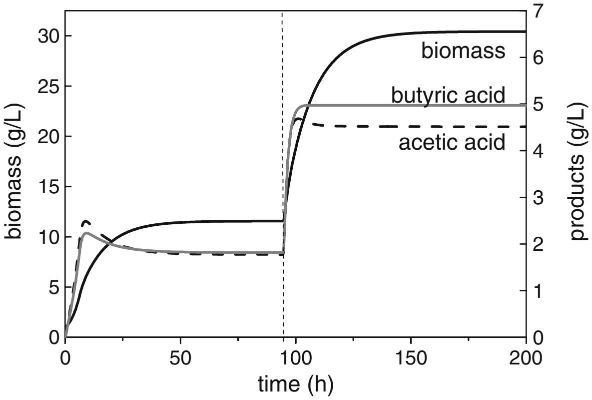

Fig. 3 Dynamics of biomass and the intermediates (acetic acid and butyric acid) concentrations in the fermentor. The *vertical dashed line* indicates the time when the flash tank separation system is turned on

Dynamic Matrix Control

The concepts of the DMC algorithm were originally presented by Cutler and Ramaker [19], and a detailed description can be found in Luyben [20].

The DMC algorithm uses a linear model to forecast the prediction horizon future output responses by calculating the required future changes in the manipulated variables that result in optimum set-point tracking for a specified performance index. A time-domain step response model (convolution model) is used to represent the process response to a step change in the input variable. The model predicts the behavior of controlled variable y at sampling time k, based on the present measured value $\left(y_0^{\mathrm{meas}}\right)$, the coefficients of the step response model, and past changes in the manipulated variable $(\Delta m_k)^{\mathrm{old}}$. The DMC algorithm tries to minimize the squared sum of the deviations between the predicted output in the closed loop form and the set-point values at control horizon future sampling time periods. The solution for a SISO system is given by

$$(\Delta m)^{\mathrm{new}} = \left[A^{\mathrm{T}}A + f^2 I\right]^{-1} A^{\mathrm{T}}\left[y^{\mathrm{set\ point}} - y_{\mathrm{OL},i}\right] \quad (8)$$

where $(\Delta m_k)^{\mathrm{new}}$ is the vector of optimal values of future changes in the manipulated variables on the control horizon. Equation 8 depends on the dynamic matrix A, which is based on the convolution model coefficients, on the weighting factor f, and on the difference between the set point value ($y^{\mathrm{set\ point}}$) and that predicted by the convolution

Table 1 Operating conditions of the continuous flash fermentation process.

Parameter	Value	Unit
V	300	m^3
F_0	100	m^3/h
S_0	142.9	g/L
F_{PU}	25	m^3/h
F_c	500	m^3/h
T_{ferm}	37	°C
T_{flash}	37	°C
P_{flash}	6.50	kPa

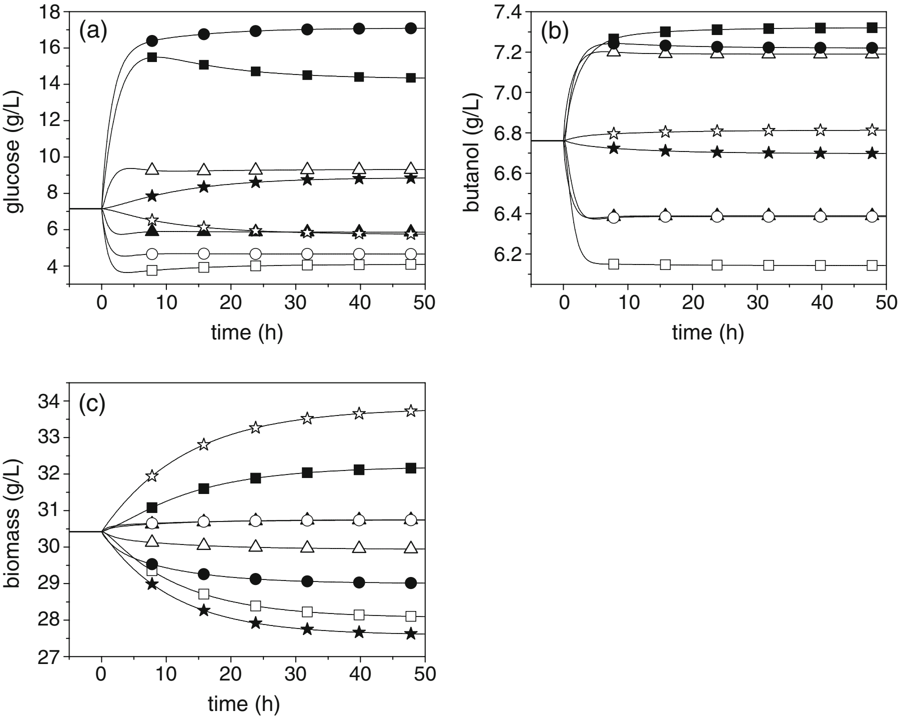

Fig. 4 Dynamics of substrate (**a**), butanol (**b**), and biomass (**c**) concentrations in the fermentor after perturbations of ±10% in the manipulated variables (F_0, F_{PU}, and F_c) and in the inlet substrate concentration (S_0) around the steady-state given by the operating conditions of Table 1. *Filled circle* $+F_0$, *empty circle* $-F_0$, *filled square* $+S_0$, *empty square* $-S_0$, *filled triangle*, $+F_c$, *empty triangle* $-F_c$, *filled star* $+F_{PU}$, empty star $-F_{PU}$

model ($y_{OL,i}$). The tuning parameters of the DMC controller are the prediction horizon (N_P), the control horizon (N_C), and the weighting factor (f). This procedure can be extended for the MIMO case with little conceptual effort.

In this study, the DMC algorithm was implemented in a Fortran program. The procedure proposed by Maurath et al. [21] was used for initial estimation of the control parameters (N_P, N_C, and f). With initial DMC tuning parameters, the refined tuning procedure was basically concerned with varying f to adjust the behavior of controlled variables, avoiding oscillations. Values of tuned parameters were found to be in a range of up to ±40% of the initial estimation. Control parameters were tuned for the cases of regulatory control, since load regulation is far more important than set-point response, considering that, in continuous processes, load changes are more frequent and can be severe.

Table 2 Effects of the inputs on process outputs.

Output variables	Input variables			
	S_0	F_0	F_{PU}	Fc
X				
S				
P_{but}				

Black area means that the input strongly influences the output (response variation greater than 30%), the *white area* means that the influence is weak (response variation up to 5%), and the *gray area* means that the input has a medium influence on the output (response variation between 5% and 30%)

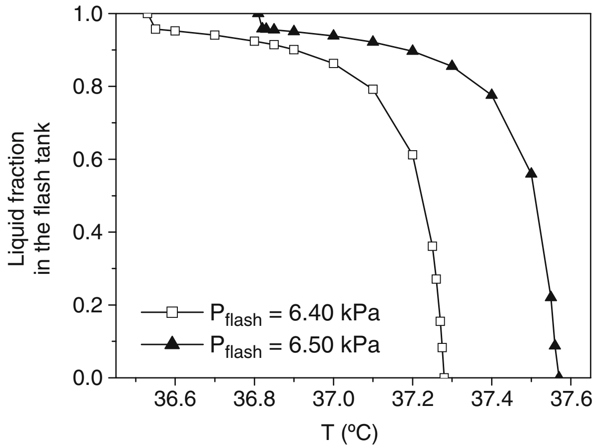

Fig. 5 Effect of P_{flash} and T_{flash} on the liquid fraction in the flash tank considering the operating conditions of Table 1

Proportional-Integral Controller

Even with the continuous development of the advanced controllers and their growing implementation in industrial processes, nowadays, it is more common to use classical control techniques, such as the PI controller, due to relatively low cost and ease of implementation.

The PI control algorithm is given by Eq. 9:

$$\Delta m_n = K_{\mathrm{c}} \left(x_n + \frac{\Delta t}{\tau_i} \sum_{k=1}^{n} x_k \right) \tag{9}$$

where Δm_n is variation of the manipulated variable, x_n is the controlled variable error (set-point minus measured value) at time n, K_{c} is the proportional gain, τ_i is the integral gain, and Δt is the sampling time.

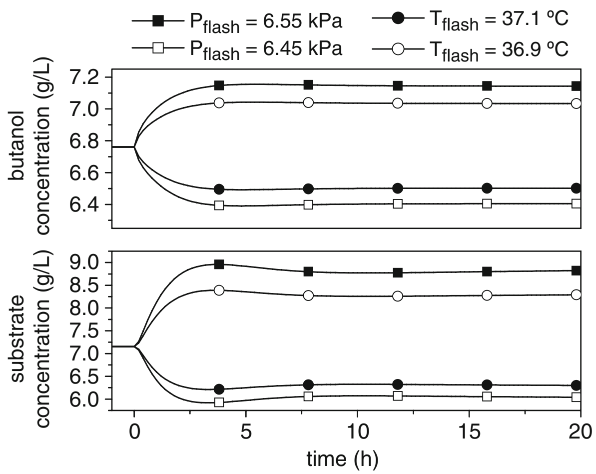

Fig. 6 Dynamics of substrate and butanol concentrations in the fermentor after perturbations of P_{flash} and T_{flash}

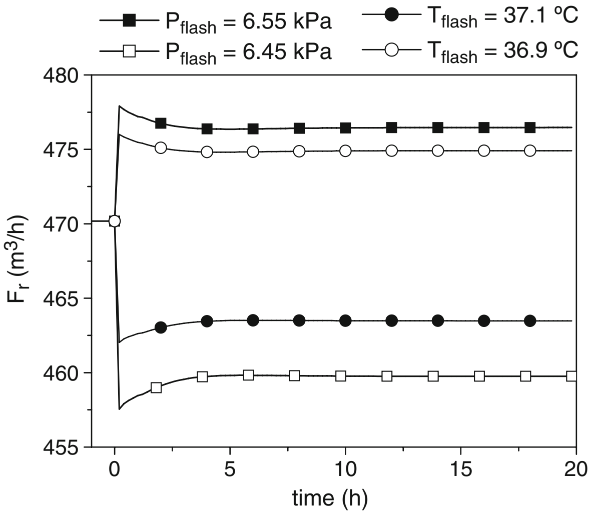

Fig. 7 Dynamics of liquid flow rate (F_r) in the flash tank after perturbations of P_{flash} and T_{flash}

The initial estimation of the parameters of the PI control (K_c and τ_i) was obtained according to the Ziegler–Nichols method [22] for the cases of regulatory control.

Results and Discussion

Dynamic Behavior of the Process

To choose the best control structures for the process, its open-loop dynamic behavior was investigated. The objective was to determine how the output variables are influenced by changes in the inputs (manipulated variables and possible disturbances). This was done by

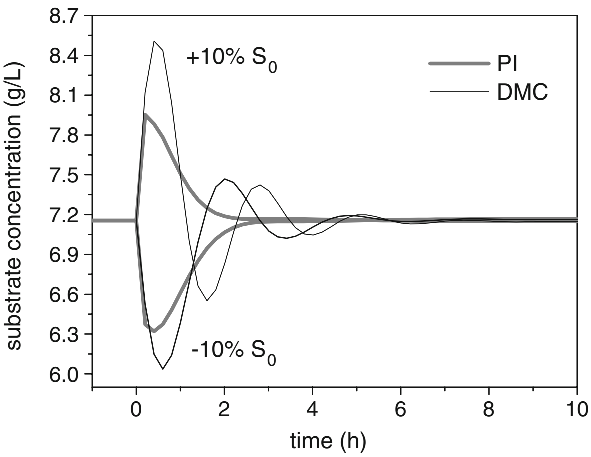

Fig. 8 Regulatory control for substrate concentration for step disturbances of ±10% in feed substrate concentration

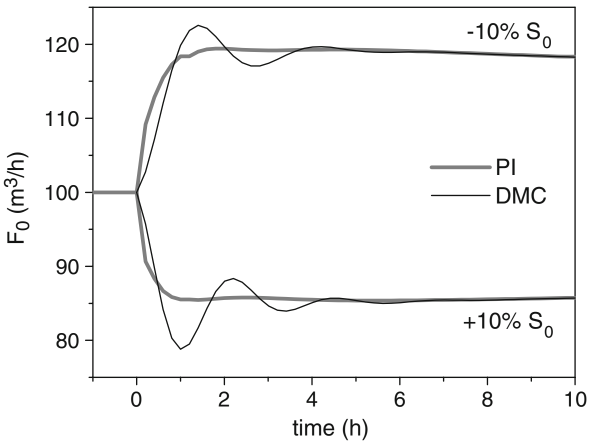

Fig. 9 Control action (manipulation of F_0) in relation to Fig. 8

changing the values of the various input variables and observing the change of the output variables with time.

The outputs of the process are the concentrations in the fermentor of the following variables: substrate (S), butanol (P_{but}), and biomass (X). The input variables considered for manipulation are inlet flow rate (F_0), purge flow rate (F_{PU}), and the feed flow of the flash tank (F_c). The input variable considered as possible load disturbances is the inlet substrate concentration (S_0). For the alterations of F_0, F_{PU} was maintained as being 25% of F_0 in order to keep the fermentor volume constant.

Figure 4 shows the output variables (S, P_{but}, and X) as functions of time after step perturbation of ±10% in the manipulated variables around the steady-state given by the operating conditions of Table 1. The system presents a dynamic behavior of first-order, with non-linearities caused by the product of output variables in the kinetic model. The dynamics of the substrate and butanol concentrations are faster than the dynamics of the biomass, as can be seen by the differences in the shape of the curves in Figs. 4a and b compared to c. The formers take approximately one third of the time biomass concentration needs to achieve a new steady state. No delays or inverse responses were observed.

Based on the responses of the process (magnitude of the variations), a table of the effects of the inputs on the outputs can be constructed. In Table 2, the black area means that the input strongly influences the output (response variation greater than 30%), the white area means that the influence is weak (response variation up to 5%), and the gray area means that the input has a medium influence on the output (response variation between 5% and

Table 3 Effects of the regulatory control of substrate concentration on other variables of the process.

Control	–	–	–	PI	PI	DMC	DMC
Perturbation in S_0 (%)	0	+10	–10	+10	–10	+10	–10
X (g/L)	30.4	31.9	28.5	33.1	27.7	33.1	27.7
P_{but} (g/L)	6.76	7.31	6.15	6.88	6.62	6.88	6.62
Butanol productivity (g/L.h)	9.21	10.2	8.17	9.15	9.30	9.15	9.30
Butanol yield (%)	19.3	19.5	18.8	20.0	18.7	20.0	18.7
Substrate conversion (%)	95.0	90.5	96.9	95.4	94.4	95.4	94.4

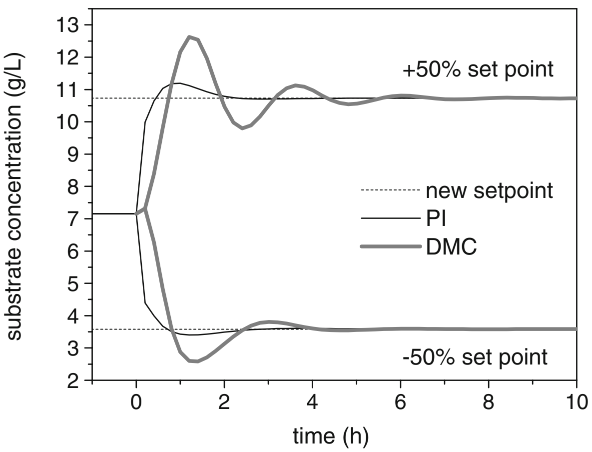

Fig. 10 Supervisory control for substrate concentration for changes of ±50% in the set point

30%). This analysis was used to determine the best control structures of the process, to know that substrate concentration (S) can be controlled by manipulating F_0, that control of butanol concentration can be achieved by manipulation of F_0 or F_c, that biomass concentration can be controlled by the manipulation of F_{PU}, and disturbances in S_0 have medium (X and P_{but}) and strong (S) influences on the output variables. Note that changes in F_0 had weak effect on biomass concentration because the purge flow was tied to F_0 (F_{PU} was 25% of F_0). Then, due to the opposite effects of F_0 and F_{PU} on X, these effects were counterbalanced.

In the previous analysis, the temperature and pressure of the flash tank were considered constant. The values of these two variables in combination with the concentrations of the components determine the conditions for the liquid–vapor equilibrium.

The concentration of the components in the flash tank varies according to operating conditions of the fermentor. On the other hand, variations of temperature and pressure can break the thermodynamic equilibrium and consequently interrupt the recovery of butanol from the fermentation broth. Considering the process in steady state (operating conditions

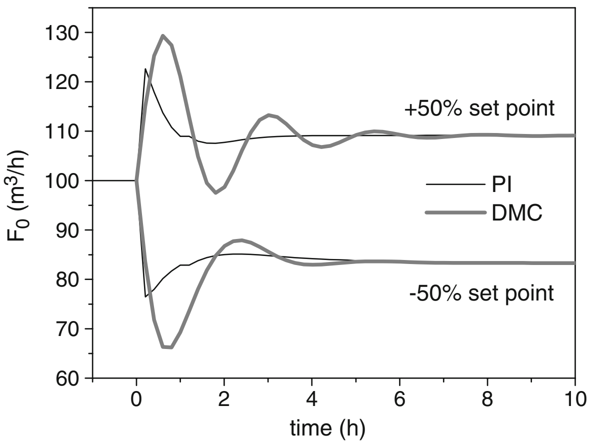

Fig. 11 Control action (manipulation of F_0) in relation to Fig. 10

Table 4 Parameters of the controllers—butanol concentration control.

Controller	PI	PI	PI (two SISO loops)	DMC	DMC	DMC-MISO
Manipulated variable	F_0	F_c	F_0 and F_c	F_0	Fc	F_0 and F_c
parameters	K_c=40	K_c=363.6	$K_c = 30$ $\tau_i = 2.5(F_0)$	N_P=3	N_P=4	N_P=3
	τ_i=3	τ_i=1	$Kc = 30$ $\tau_i = 2.5(Fc)$	N_C=2	N_C=3	N_C=5
				f=0.33	f=0.06	f=0.0033 (F_0)
						$f = 4.3 \times 10^{-4}(F_c)$

of Table 1), values of pressure or temperature out of the following ranges $6.30 \leq P_{\text{flash}} \leq 6.56\,\text{kPa}(\text{for}\,T_{\text{flash}} = 37.0^{\text{o}}\text{C})$ and $36.8 \leq T_{\text{flash}} \leq 37.5^{\text{o}}\text{C}(\text{for}\,P_{\text{flash}} = 6.50\,\text{kPa})$ caused the break of the equilibrium. Note that temperature and pressure ranges were obtained from the flash calculation (Eq. 7), and they limit the conditions under which two phases (liquid and vapor) were observed in the flash tank.

Figure 5 shows that the operation of the flash tank is very complex, since there is a nonlinear relationship between the liquid fraction and T_{flash} for a given P_{flash}. These characteristics show that an effective maintenance of the conditions (P_{flash} and T_{flash}) of the flash tank must be ensured for the proper operation of the flash fermentation.

Variations in P_{flash} and T_{flash} have effects on substrate and butanol concentrations (Fig. 6) and on the liquid flow rate leaving the flash tank (F_r) (Fig. 7). The response to the perturbations followed the dynamics of a first-order system, being nonlinear the responses of substrate concentration and F_r. The response of the latter is faster than the others, and values next to steady state are achieved very fast (12 min) after perturbations, so that only slight changes in F_r values are noticed after this initial period. Responses of the concentrations take approximately 4 h to be close to steady state.

Despite of the effects of P_{flash} and T_{flash} on the system, these variables cannot be manipulated for the control of the process outputs (S, P_{but}, and X), since alterations of these variables could break the liquid–vapor equilibrium, as also reported by Atarassi [23] when studying the use of the flash technology for the ethanol fermentation.

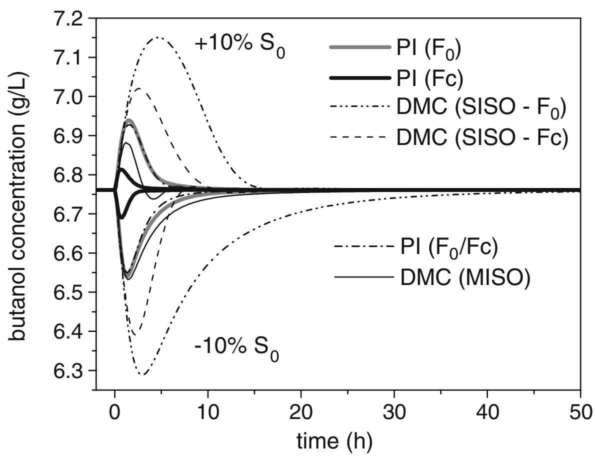

Fig. 12 Regulatory control for butanol concentration for step disturbances of ±10% in feed substrate concentration

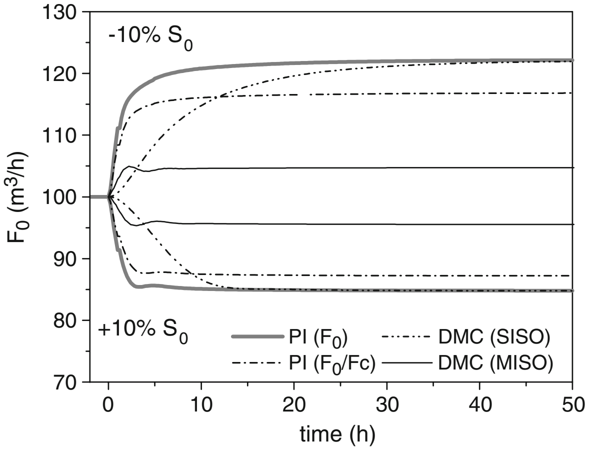

Fig. 13 Control action (manipulation of F_0) in relation to Fig. 12

On the other hand, perturbations of T_{flash} are expected to happen, and given the narrow range of values that this variable can assume, it makes necessary the manipulation of P_{flash} in order to keep constant the amount of butanol recovered in the flash tank. In this regard, the liquid flow rate of the flash tank (F_r) can be used as a controlled variable, given the effect of T_{flash} on the proportion between the liquid and vapor fractions in the flash tank and the fast response F_r presents for alterations in T_{flash} and P_{flash}.

Process Control

Substrate Concentration Control

Based on the results of the dynamic behavior study, a SISO loop was used to control substrate concentration in the fermentor in the face of perturbations of the inlet substrate concentration (S_0). The inlet flow rate (F_0) was chosen as the manipulated variable. For

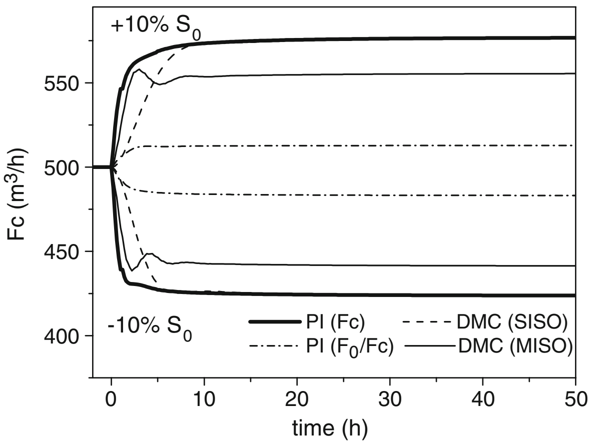

Fig. 14 Control action (manipulation of *Fc*) in relation to Fig. 12

Table 5 Effects of the regulatory control of butanol concentration on other variables of the process.

Control	PI						DMC					
Manipulated variable	F_0	F_0	F_c	F_c	F_0/F_c	F_0/F_c	F_0	F_0	F_0	F_c	F_0/F_c	F_0/F_c
perturbation in S_0 (%)	+10	−10	+10	−10	+10	−10	+10	−10	+10	−10	+10	−10
X (g/L)	33.2	27.4	33.1	27.6	33.2	27.5	33.1	27.4	33.0	27.7	33.1	27.7
S (g/L)	6.19	8.93	9.57	5.45	6.62	7.92	6.22	8.82	9.61	5.43	8.43	6.03
butanol productivity (g/L.h)	8.87	9.71	10.3	8.15	9.11	9.36	8.87	9.70	10.3	8.15	9.89	8.50
butanol yield (%)	20.0	18.5	19.6	19.0	19.9	18.7	20.0	18.5	19.6	19.0	19.8	18.9
substrate conversion (%)	96.1	93.0	93.9	95.8	95.8	93.8	96.0	93.1	93.9	95.8	94.6	95.3

alterations of F_0, the value of F_{PU} was maintained as being 25% of F_0 in order to keep the fermentor volume constant.

A step change of +10% was made in S_0 in order to tune the parameters of the controllers, which were adjusted to $K_c = 9.76\,\mathrm{m}^3/(\mathrm{g/L})$ and τ_i=1 h (PI) and N_P=5, N_C=3, and f=0 (DMC). The sampling time was chosen to be 12 min, considering that a high performance liquid chromatography (HPLC) could be used to measure substrate concentration online.

In the first test of the performance of the controllers, step changes of ±10% were made in S_0 (regulatory control) at time t=0 h, i.e., S_0 values were instantaneously changed at t=0 h to a new value and kept constant at this new value indefinitely. In open loop, the substrate concentration changed from 7.15 to 14.3 and 4.09 g/L (Fig. 4) due to positive and negative disturbances, respectively. Figure 8 shows that both controllers were able to keep the controlled variable in the set-point value (7.15 g/L), by smoothly manipulating F_0 (Fig. 9). However, the DMC controller showed oscillations with a higher overshoot and required longer to return to the set point. Table 3 shows that, with the control loop, other important variables of the process were little altered after the perturbations. Otherwise, without the control loop, substrate conversion and butanol productivity suffered significant drops. After the perturbations of +10%, conversion dropped from 95% to 90.5%, and for the case of −10%, productivity changed from 9.21 to 8.17 g/L h.

The performance of the controllers for the servo problem was tested by making step changes of ±50% in the set-point value. Figures 10 and 11 present the results for the controlled and manipulated variables, respectively. It can be seen that the controllers presented good performance for the servo problem and that the DMC controller showed the same problems observed in the regulatory control.

Butanol Concentration Control

According to the dynamic analysis, control of butanol concentration can be achieved by manipulation of F_0 or F_c. Thus, besides single loops (control of P_{but} by F_0 or F_c), other control strategies were tested: DMC-MISO (control of P_{but} by manipulation of F_0 and F_c) and, for the PI controller, implementation of two concurrent SISO loops (P_{but}/F_0 and P_{but}/F_c).

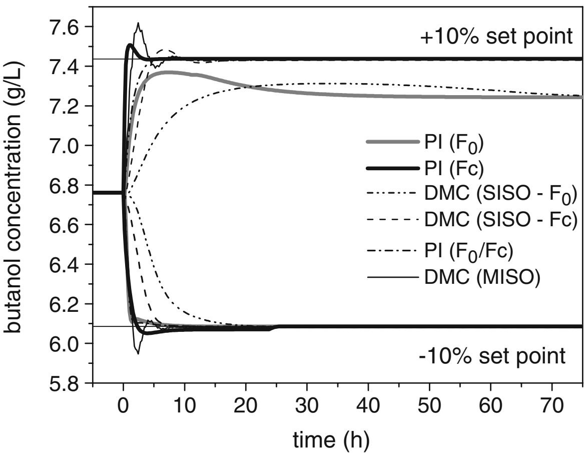

Fig. 15 Supervisory control for butanol concentration for changes of ±10% in the set point

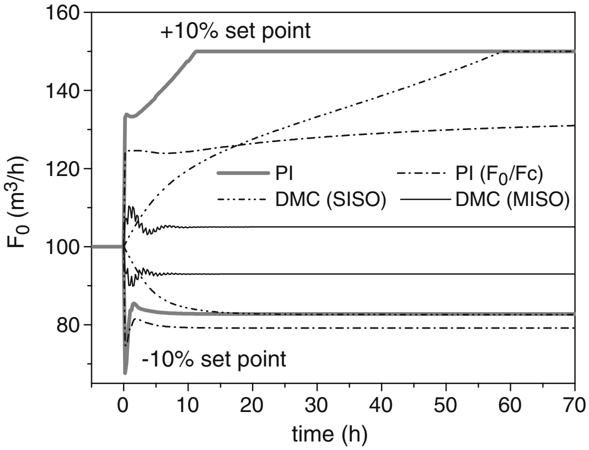

Fig. 16 Control action (manipulation of F_0) in relation to Fig. 15

For the loops with manipulation of F_0, F_{PU} was maintained as being 25% of F_0 in order to keep the fermentor volume constant.

A step change of +10% was made in S_0 in order to tune the parameters of the controllers, whose values are shown in Table 4. The sampling time was chosen to be 12 min, considering once more that an HPLC could be used to measure butanol concentration on-line.

In the first test of the performance of the controllers, step changes of ±10% were made in S_0 (regulatory control) at t=0 h. In open loop, the butanol concentration changed from 6.76 to 7.31 and 6.15 g/L (Fig. 4) due to positive and negative disturbances, respectively.

Figure 12 shows that the control strategies were able to keep the controlled variable in the set-point value (6.76 g/L) by smoothly manipulating F_0 and/or F_c (Figs. 13 and 14). The strategy with quicker response and lower overshoot was the one with the manipulation of F_c by the PI controller. On the other hand, one of the advantages of the strategies that combined the manipulation of F_0 and F_c is that the regulation of the butanol concentration was achieved with narrower variations of these variables. The other advantage is that

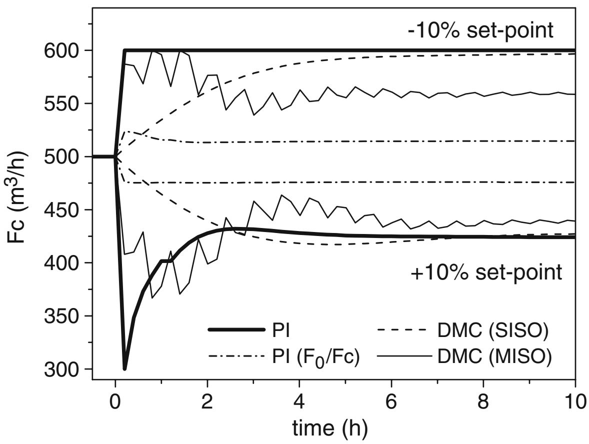

Fig. 17 Control action (manipulation of F_c) in relation to Fig. 15

Table 6 Effects of the servo control of butanol concentration on other variables of the process.

Control	PI						DMC					
Manipulated variable	F_0	F_0	F_c	F_c	F_0/F_c	F_0/F_c	F_0	F_0	F_0	F_c	F_0/F_c	F_0/F_c
Set point (%)	+10	−10	+10	−10	+10	−10	+10	−10	+10	−10	+10	−10
X (g/L)	25.9	30.8	29.6	31.0	27.2	30.9	25.9	30.8	29.6	31.0	29.4	31.0
S (g/L)	36.1	3.49	11.1	5.06	28.1	3.69	36.1	3.48	11.0	5.05	13.0	4.50
Butanol productivity (g/L h)	11.3	7.61	9.23	9.12	10.7	7.83	11.3	7.60	9.23	9.12	9.57	8.64
Butanol yield (%)	18.6	19.3	19.4	19.1	17.0	19.3	15.8	19.3	19.4	19.1	19.1	19.2
Substrate conversion (%)	74.7	97.5	92.2	96.4	80.3	97.4	77.9	97.6	92.3	96.5	90.9	96.9

variations in substrate conversion and butanol productivity (Table 5) were lower (up to 1.3% and 7.7%, respectively) than those verified in the strategies with non-combined manipulations of F_0 and F_c (up to 2.1% and 11.8%). Note that conversion and productivity variations were obtained from the difference between values before perturbation (Table 3) and after control action (Table 5).

The performance of the controllers for the servo problem was tested by making step changes of ±10% in the set-point value. Figure 15 presents the results for the controlled variable and the control actions in F_0 and F_c are in Figs. 16 and 17, respectively. The strategies, in which only F_0 was manipulated, were not able to increase the butanol concentration in the fermentor to the desired level. For these cases, variations of F_0 until and above its upper limit (150 m^3/h) were not able to increase butanol concentration to the new set point (+10%). Thus, the servo control was successful only in the strategies in which F_c was exclusively manipulated or combined with changes in F_0. The advantage of the combination is that the control was carried out with narrower variations of these variables.

As observed for the regulatory control, the strategy with better performance, i.e., with quicker response, lower overshoot, and without oscillations, was the manipulation of F_c by the PI controller. For this case, F_c reached the upper limit (600 m^3/h) for the alteration in −10% of the set point. It is also important to mention that, with this strategy (manipulation of F_c), variations in substrate conversion and butanol productivity (Table 6) were lower (2.9% and 1.0%, respectively) than those verified in the other strategies (up to 21.4% and 22.7%). Note that conversion and productivity variations were obtained from the difference between values before perturbation (Table 3) and after control action (Table 6).

Concurrent Regulatory Control of Substrate and Butanol Concentrations

For the concurrent regulatory control of substrate and butanol concentrations after perturbations in S_0, two control strategies were tested. For the PI controller, two SISO loops were implemented: control of S and P_{but} by manipulation of F_0 and F_c, respectively. For the DMC controller, a MIMO loop was considered for the same controlled and manipulated variables.

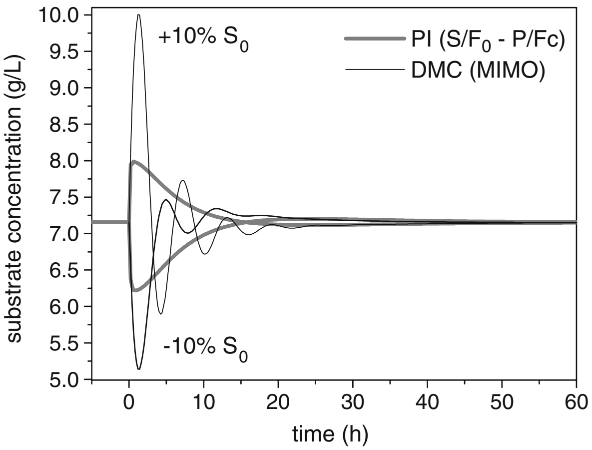

Fig. 18 Regulatory control for substrate concentration for step disturbances of ±10% in feed substrate concentration—concurrent control

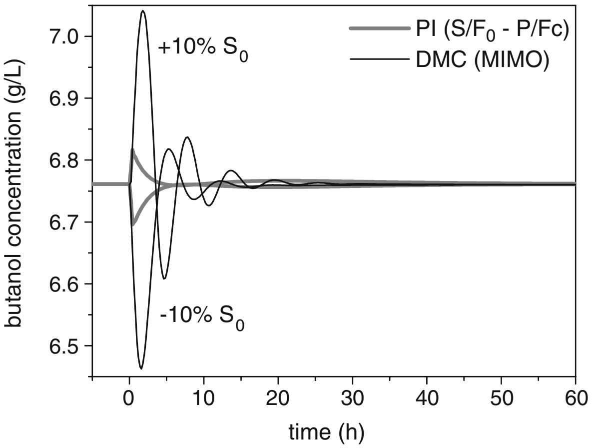

Fig. 19 Regulatory control for butanol concentration for step disturbances of ±10% in feed substrate concentration—concurrent control

A step change of +10% was made in S_0 in order to tune the parameters of the controllers, which were adjusted to $K_{c_1} = 10\ \mathrm{m}^3/(\mathrm{g/L})$ and $\tau_i 1 = 8\,\mathrm{h}(\mathrm{PI} - F_0)$; $K_{c_2} = 300\ \mathrm{m}^3/(\mathrm{g/L})$ and $\tau_{i_2} = 5\,\mathrm{h}(\mathrm{PI} - F_c)$ and N_P=4, N_C=3, and f_1=0.18 (F_0) and f_2=0.022 (F_c). The sampling time was 12 min for both concentrations.

Figures 18 and 19 show that both controllers were able to keep the controlled variables in the set-point values. The control actions can be seen in Figs. 20 and 21. The PI controller had better performance, since the DMC controller showed oscillations with a higher overshoot and demanded longer to bring the process back to the set point.

The advantage of controlling concurrently the substrate and butanol concentrations over the previous strategies is that both substrate conversion and butanol productivity (Table 7) were kept near (variations of 0.6% and 1.8%, respectively) to their values before perturbation (Table 3). Besides, control was carried out with low variations of F_0 and F_c (up to +12.4 m^3 and −31.8 m^3, respectively).

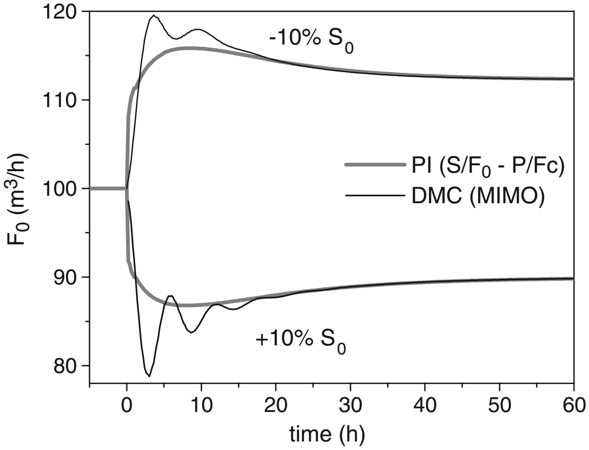

Fig. 20 Control action (manipulation of F_0) in relation to Figs. 18 and 19

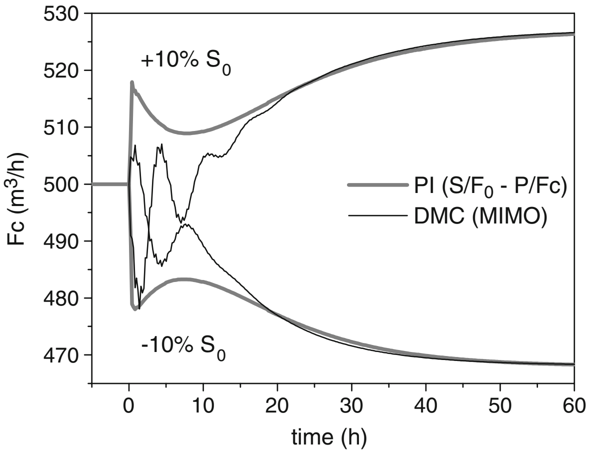

Fig. 21 Control action (manipulation of F_c) in relation to Figs. 18 and 19

Flash Tank Control

In order to keep the flash tank operating properly, i.e., to ensure the liquid–vapor equilibrium in the face of disturbances in its temperature (T_{flash}), a SISO loop was used to control the pressure (P_{flash}) based on the control of the liquid flow rate of the flash tank (F_r).

A step change of +0.1 °C was made in T_{flash} in order to tune the parameters of the controllers, which were adjusted to $K_c = 2.5 \times 10^{-3}\mathrm{kPa}/(\mathrm{m}^3)$ and $\tau_i = 1.0 \times 10^{-3}\mathrm{h(PI)}$ and N_P=3, N_C=1 and f=110 (DMC). The sampling time was chosen to be 3.6 s.

To test the performance of the controllers (regulatory control), a saw-tooth temperature oscillation was applied to the flash tank (Fig. 22). P_{flash} was manipulated according to the variations in F_r (Fig. 22), and both controllers (PI and DMC) were equally able to maintain the vapor–liquid separation in the flash tank. The control actions followed a linear relationship with the oscillations in T_{flash} (the operation of the flash tank between 35 and 39 °C was associated to a pressure range of 5.85 and 7.22 kPa), and kept the butanol, substrate, and biomass concentrations in the fermentor oscillating very close to their set points (Fig. 23).

The last test consisted in controlling the concentrations in the fermentor and the pressure in the flash tank considering simultaneous disturbances in S_0 and T_{flash} (Fig. 24). For this case, only the performance of the PI controller was assessed because it showed to be more suitable for controlling the process.

Table 7 Effects of the concurrent regulatory control of substrate and butanol concentration on other variables of the process.

Control	PI		DMC	
Perturbation in S_0 (%)	+10	−10	+10	−10
X (g/L)	33.2	27.6	33.2	27.6
Butanol productivity (g/L h)	9.37	9.04	9.37	9.04
Butanol yield (%)	19.9	18.8	19.9	18.8
Substrate conversion (%)	95.4	94.4	95.4	94.4

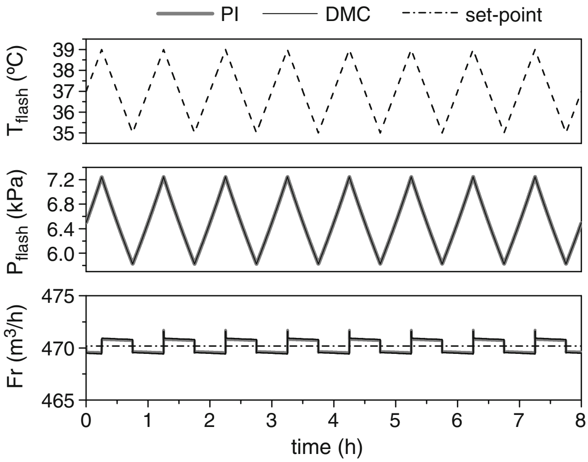

Fig. 22 Disturbances in the T_{flash}. Manipulation of P_{flash} (PI and DMC controllers) based on the variations of the liquid flow rate of the flash tank (F_r)

As the first attempt, three SISO control loops were implemented: (1) control of substrate concentration (S) by manipulation of F_0; (2) control of butanol concentration by manipulation of F_c, and (3) control of P_{flash} according to variations in F_r.

This strategy was not successful. The manipulation of F_c by loop 2 causes alterations in F_r because both flows are correlated by the mass balance in the flash tank (Eq. 6). As loop 3 interprets all variations of F_r as only being caused by alterations in T_{flash}, the pressure is not well regulated, and consequently, the thermodynamic equilibrium in the flash tank breaks. Thus, loop 2 was withdrawn from the control strategy.

With loops 1 and 3, the proper operation of the flash tank was ensured by the manipulation of P_{flash}, and the substrate concentration was kept constant in the fermentor by the manipulation of F_0 (Figs. 24 and 25). It should be noted that, since F_0 similarly

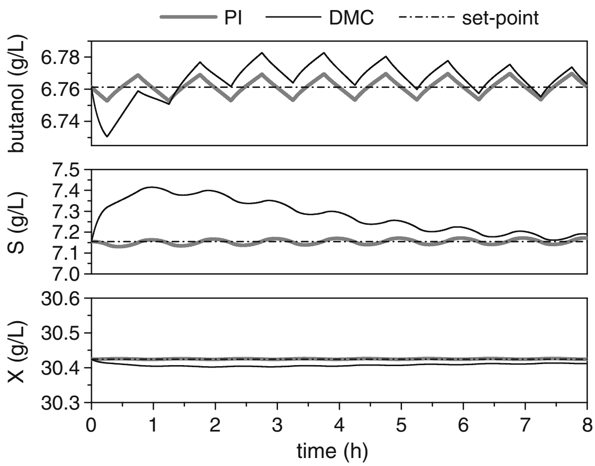

Fig. 23 Butanol, substrate, and biomass concentrations in the fermentor. Process under the disturbances shown in Fig. 22 and the action of the PI and DMC controllers

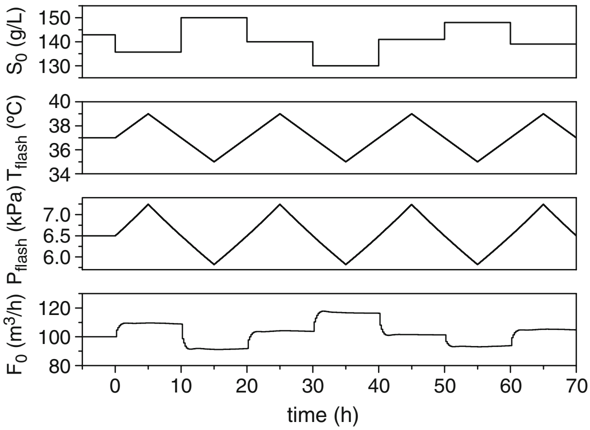

Fig. 24 Disturbances in S_0 and T_{flash} and respective manipulations of F_0 and P_{flash} (PI controller)

influences the butanol concentration (Fig. 4), the manipulation of the former resulted in an indirect satisfactory control of the latter.

In order to carry out the direct control of the butanol concentration (loop 2), it would be necessary to substitute loop 3 by a feedforward control of P_{flash} based on the measure of the perturbations in T_{flash}, thus avoiding the conflict between loops 2 and 3.

Conclusions

With the use of a mathematical model, it was possible to assess the dynamic behavior of the flash fermentation process for the production of butanol. This study was necessary to elaborate suitable control strategies able to deal with disturbances related to the process. Disturbances in the feed substrate concentration and in the temperature of the flash tank had

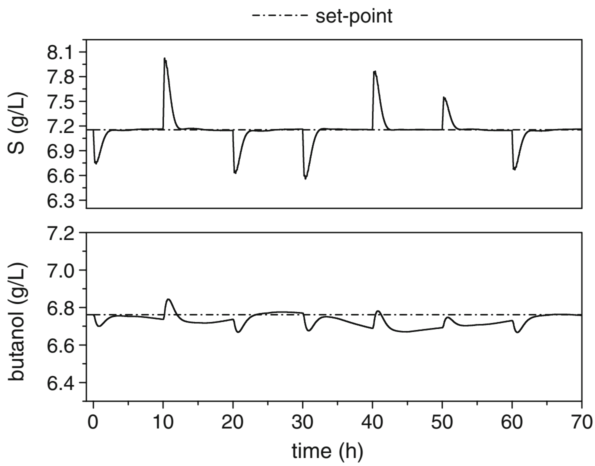

Fig. 25 Butanol and substrate concentrations in the fermentor. Process under the disturbances shown in Fig. 24 and the action of the PI controller

severe impacts on the process. While the former can cause significant drops in the substrate conversion and butanol productivity, with the latter the butanol recovery in the flash tank can be interrupted.

Both controllers (PI and DMC) were able to regulate the operating conditions in order to accommodate the perturbations with the lowest possible alterations in the process outputs. However, the performance of the PI controller was superior because it showed quicker responses without oscillations.

There is an incompatibility between the feedback loops that control the butanol concentration in the fermentor and the pressure in the flash tank. For this reason, it is suggested the substitution of the latter loop (control of pressure by manipulation of the flash tank inlet flow rate) by a feedforward scheme to carry out the control of the pressure based on the measure of the perturbations in the flash tank temperature.

The implementation of efficient controllers in industrial-scale butanol fermentations is necessary and can be a helpful tool for today's efforts to make the biobutanol industry commercially viable.

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